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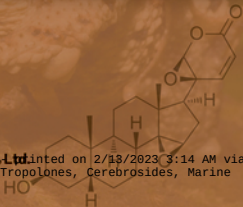
HANDBOOK OF NATURAL PRODUCTS

**VOLUME 6: STEROIDS, TROPOLONES, CEREPROSIDES,
MARINE NATURAL PRODUCTS, AMINO ACIDS, AND
SUGARS**

Edited by Hailin Qin, Dequan Yu



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^1H NMR Handbook of Natural Products

Volume 6: Steroids, Tropolones, Cerebrosides, Marine
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13 Steroids

13.1 Common steroids

13.1.1 Androstane-type steroids

Table 13-1-1: Compounds, MFs, and test solvents of androstane-type steroids 13-1-1 and 13-1-2.

No.	Compounds	MFs	Test solvents	Refer- ences
13-1-1	6 α -hydroxyandrosta-1,4-diene-3,17-dione	C ₁₉ H ₂₄ O ₃	CDCl ₃	[1]
13-1-2	2 α ,3 α -dihydroxyandrostan-16-one-2 β ,19-hemiketal	C ₁₉ H ₂₈ O ₄	C ₅ D ₅ N	[2]

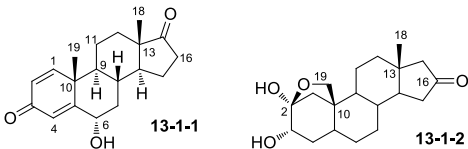


Table 13-1-2: ¹H NMR spectroscopic data of androstane-type steroids 13-1-1 and 13-1-2.

H	13-1-1	13-1-2	H	13-1-1	13-1-2
1	6.99 d(10.0)	α 2.40 d(10.8) β 2.30 d(10.4)	11	1.65 m 1.83 m	β 1.35
2	6.48 dd(1.9, 10.3)		12	1.26 m, 1.85 m	1.60
3		4.38 m	14	1.29 m	1.27
4	6.22 t(1.8)	α 2.01	15	1.59 m, 1.96 m	α 2.15 dd(7.6, 18.0) β 1.83
5		β 1.85 1.30	16	2.07 dd(10.0, 19.0) 2.45 dd(9.0, 19.0)	
6	4.49 ddd(1.7, 5.5, 11.7)	α 1.55 β 1.20	17		α 1.92 d(16.8) β 2.06 d(16.8)
7	1.10 m 2.33 ddd(3.7, 5.5, 12.0)	α 1.28 β 1.43	18	0.91 s	0.66 s
8	1.85 m	0.85	19	1.21 s	4.10 d(8.0), 3.89 d(8.0)
9	1.07 m	1.18			

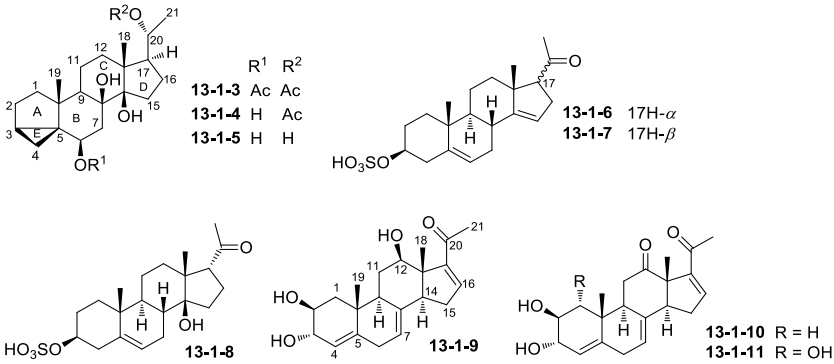
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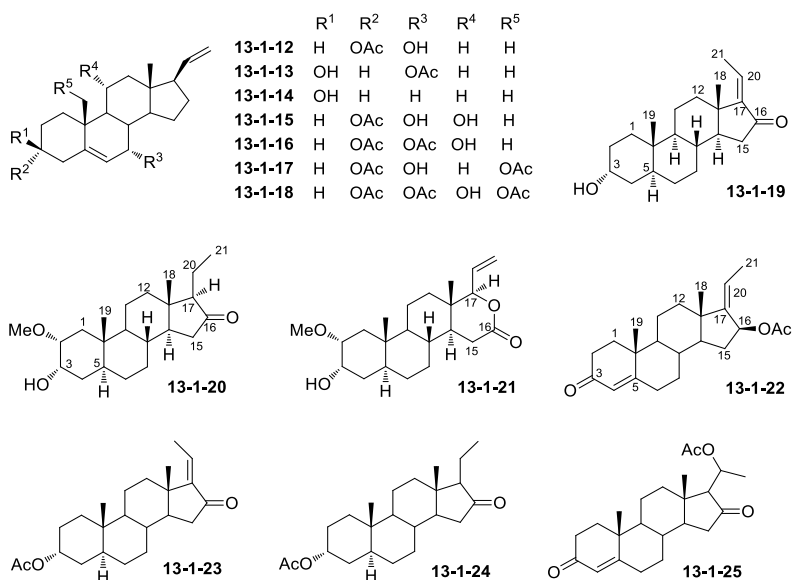
13.1.2 Pregnane-type steroids

Table 13-1-3: Compounds, MFs, and test solvents of pregnane-type steroids 13-1-3~13-1-25.

No.	Compounds	MFs	Test solvents	References
13-1-3	vladimuliecin A	C ₂₅ H ₃₈ O ₆	CDCl ₃	[3]
13-1-4	vladimuliecin B	C ₂₃ H ₃₆ O ₅	CDCl ₃	[3]
13-1-5	(3 <i>R</i> ,5 <i>R</i> ,6 <i>R</i> ,8 <i>S</i> ,9 <i>R</i> ,10 <i>R</i> ,13 <i>R</i> ,14 <i>R</i> ,17 <i>S</i> ,20 <i>R</i>)-3,5-cyclo-pregnane-6,8,14,20-tetraol	C ₂₁ H ₃₄ O ₄	CDCl ₃	[3]
13-1-6	pregna-5,14-dienolone-3- <i>O</i> -sulfate	C ₂₁ H ₃₀ O ₅ S	CD ₃ OD	[4]
13-1-7	<i>allo</i> -pregna-5,14-dienolone-3- <i>O</i> -sulfate	C ₂₁ H ₃₀ O ₅ S	CD ₃ OD	[4]
13-1-8	14β-hydroxypregn-5-enolone-3- <i>O</i> -sulfate	C ₂₁ H ₃₂ O ₆ S	CD ₃ OD	[4]
13-1-9	stizophyllin	C ₂₁ H ₂₈ O ₄	CDCl ₃	[5]
13-1-10	terresterone A	C ₂₁ H ₂₆ O ₄	CDCl ₃	[5]
13-1-11	terresterone B	C ₂₁ H ₂₆ O ₅	CDCl ₃	[5]
13-1-12	pregna-5,20-diene-3α,7α-diol 3α-acetate	C ₂₃ H ₃₄ O ₃	CDCl ₃	[6]
13-1-13	pregna-5,20-diene-3β,7α-diol 7α-acetate	C ₂₃ H ₃₄ O ₃	CDCl ₃	[6]
13-1-14	pregna-5,20-dien-3β-ol	C ₂₁ H ₃₂ O	CDCl ₃	[6]
13-1-15	pregna-5,20-diene-3α,7α,11α-triol-3α-acetate	C ₂₃ H ₃₄ O ₄ ^①	CDCl ₃	[6]
13-1-16	pregna-5,20-diene-3α,7α,11α-triol-3α,7α-diacetate	C ₂₅ H ₃₆ O ₅ ^①	CDCl ₃	[6]
13-1-17	pregna-5,20-diene-3α,7α,19-triol-3α,19-diacetate	C ₂₅ H ₃₆ O ₅	CDCl ₃	[6]
13-1-18	pregna-5,20-diene-3α,7α,11α,19-tetrol-3α,7α,19-triacetate	C ₂₇ H ₃₈ O ₇	CDCl ₃	[6]
13-1-19	(<i>E</i>)-aglawone	C ₂₁ H ₃₂ O ₂	CDCl ₃	[7]
13-1-20	aglatomin A	C ₂₂ H ₃₆ O ₃	CDCl ₃	[8]
13-1-21	aglatomin B	C ₂₂ H ₃₄ O ₄	CDCl ₃	[8]
13-1-22	16-β-acetyloxy-pregn-4-17(20)- <i>trans</i> -dien-3-one	C ₂₃ H ₃₂ O ₃	CDCl ₃	[9]
13-1-23	3-α-acetyloxy-5-α-pregn-17(20)- <i>cis</i> -en-16-one	C ₂₃ H ₃₄ O ₃	CDCl ₃	[9]
13-1-24	3-α-acetyloxy-5-α-pregnan-16-one	C ₂₃ H ₃₆ O ₃	CDCl ₃	[9]
13-1-25	20 <i>S</i> -acetyloxy-4-pregnene-3,16-dione	C ₂₃ H ₃₂ O ₄	CDCl ₃	[9]

^① The molecular formula in the main body was uncorrect, while that in the experimental section was right.



**Table 13-1-4:** ¹H NMR spectroscopic data of pregnane-type steroids **13-1-3**~**13-1-7**.

H	13-1-3	13-1-4	13-1-5	13-1-6	13-1-7
1	0.94 ddd (13.8, 7.8, 3.6)	0.92 ddd (12.0, 8.0, 3.6)	0.90 ddd (12.8, 7.6, 3.6)	1.97 br ddd (13.6, 3.7, 3.6)	1.90 br dddd (13.6, 3.7, 3.6, <0.5)
	0.92 ddd (13.8, 6.0, 5.4)	0.86 ddd (12.0, 6.0, 4.8)	0.93 ddd (12.8, 5.6, 4.0)	1.13 ddd (13.6, 13.5, 3.7)	1.08 ddd (13.6, 13.5, 3.7)
2	1.62 dm(7.8)	1.62 dm(8.0)	1.62 dm(7.6)	2.08 dddd (13.0, 3.7, 3.6, 2.6)	2.05 ddddd (13.0, 4.8, 3.7, 3.6, 2.6)
				1.67 dddd (13.5, 13.0, 11.6, 3.6)	1.63 dddd (13.5, 13.0, 11.6, 3.6)
3	1.20 dddd (7.8, 4.8, 4.2, 3.6)	1.11 ddt (8.0, 4.8, 4.0)	1.08 ddt (8.0, 4.8, 4.0)	4.16 dddd/tt (11.6, 11.6, 4.8, 4.8)	4.15 dddd/tt (11.6, 11.6, 4.8, 4.8)
4	0.59 t(4.8) 0.64 dd(7.8, 4.8)	0.44 dd(8.0, 4.8) 0.65 t(4.8)	0.46 dd(8.0, 4.8) 0.64 t(4.8)	2.56 ddd (13.3, 4.8, 2.6)	2.55 ddd (13.3, 4.8, 2.6)
				2.36 ddddd (13.3, 11.6, 2.8, 2.5, 2.1)	2.35 ddddd (13.3, 11.6, 2.8, ca. 2, ca. 2)

Table 13-1-4 (continued)

H	13-1-3	13-1-4	13-1-5	13-1-6	13-1-7
6	4.82 t(3.0)	3.50 brs	3.46 brs	5.45 br ddd (4.6, 2.8, 2.3, <0.5)	5.45 br ddd (4.6, 2.8, 2.7, <0.5)
7	2.50 dd(15.0, 3.0) 1.68 dd(15.0, 3.0)	2.34 dd(14.4, 3.0) 1.52 dd(14.4, 3.2)	2.34 dd(14.4, 2.8) 1.53 dd(14.4, 3.2)	2.22 dddd (16.9, 5.5, 4.6, 2.1) ca. 2.10	2.18 dddd (16.9, 5.5, 4.6, 2.1) ca. 2.10
8				ca. 2.10	ca. 2.10
9	1.30 m	1.29 m	1.30 m	1.03 ddd (12.2, 11.6, 3.7)	0.94 ddd (12.2, 11.6, 3.7)
11	1.31 m	1.33 m	1.32 m	1.72 dddd (12.2, 6.1, 3.7, 1.5) ca. 1.62	ca. 1.71 ca. 1.62
12	1.32 m 1.69 m	1.33 m 1.68 m	1.33 m 1.68 m	ca. 2.21 ca. 1.55	ca. 2.21 1.23 ddd (17.1, 12.3, 7.2)
15	1.56 dm(10.2)	1.60 dm(10.2)	1.64 dm(10.0)	5.21 br ddd (2.3, 2.2, 1.8, <0.5)	5.17 ddd (2.3, 2.3, 2.2)
16	1.80 m	1.80 m	1.78 m	α 2.18 β 2.72 dddd (16.0, 10.1, 4.3, 1.8)	2.38 dddd (16.6, 8.8, 3.6, 2.2)
					2.64 dddd (16.6, 5.4, 2.5, 2.3)
17	1.83 dm(9.0)	1.74 dm(9.2)	1.78 dm(9.0)	3.01 br dd/m (10.1, 8.2, <0.5)	3.15 dd(8.8, 5.4)
18	1.15 s	1.18 s	1.38 s	0.86 s	1.35 s
19	1.12 s	1.17 s	1.19 s	1.05 brs	1.07 brs
20	4.97 dq(9.0, 6.0)	4.97 dq(9.2, 6.4)	3.87 dq(9.0, 6.0)		
21	1.16 d(6.0)	1.16 d(7.2)	1.26 d(6.4)	2.16 s	2.16 s
OAc	2.09 s(6-OAc) 2.01 s(20-OAc)	2.00 s(20-OAc)			

Table 13-1-5: ^1H NMR spectroscopic data of pregnane-type steroids **13-1-8~13-1-11**.

H	13-1-8	13-1-9	13-1-10	13-1-11
1	1.92 br ddd(13.7, 3.7, 3.6, <0.5) 1.14 ddd(13.7, 13.5, 3.7)	α 2.07 dd(13, 4) β 1.36 dd(12, 13)	α 1.93 dd(13.7, 3.8) β 1.39 dd(12.6, 13.7)	β 3.45 d(10.4)
2	2.08 dddd(13.0, 4.8, 3.7, 3.6, 2.6) 1.64 dddd(13.5, 13.0, 11.6, 3.6)	α 3.59 ddd(12, 8, 4)	α 3.56 ddd(3.8, 8.0, 12.6)	α 3.58 dd(7.0, 10.4)
3	4.15 dddd/tt(11.6, 11.6, 4.8, 4.8)	β 4.06 m	β 4.07 ddd(1.6, 3.6, 8.0)	β 4.10 m
4	2.55 ddd(13.0, 4.8, 2.3) 2.35 dddd(13.0, 11.6, 2.8, 2.5, 2.1)	5.24 br s	5.30 t(1.6)	5.26 br s
6	5.43 br ddd(4.6, 2.8, 2.3, <0.5)	α 2.47 m β 2.99 br d(18)	α 2.61 br dd(6, 18) β 3.10 br d(18)	α 2.54 br dd(4.1, 18.8) β 3.0 br d(18.8)
7	2.25, 1.85	5.35 m	5.51 m	5.53 m
8	1.71 ddd(11.5, 10.8, 5.5)			
9	1.22 ddd(12.2, 11.5, 4.7)	α 2.31 m	α 2.71 m	α 2.82 m
11	ca. 1.55 ca. 1.46	α 2.03 m β 1.59 ddd(13, 12, 10)	α 2.43 dd(11.5, 5.2) β 2.75 t(11.5)	α 2.98 dd(13.7, 5.2) β 2.75 t(13.7)
12	ca. 1.55, ca. 1.46	α 3.89 dd(10, 5)		
14		α 2.20 m	α 2.50 br dd(11.5, 1.6)	α 2.40~2.48 m (ov)
15	2.13 ddd(13.2, 10.6, 9.0) 1.74 ddd(13.2, 10.6, 2.3)	α 2.37 m β 2.52 m	α 2.47 m	α 2.40~2.48 m (ov)
16	2.03 dddd(13.0, 10.6, 10.6, 9.4) ca. 1.88 dddd(13.0, 9.0, 4.6, 2.3)	7.00 br s	6.64 br s	6.64 br s
17	2.97 dd(9.4, 4.6)			
18	0.98 s	0.79 s	1.24 s	1.22 s
19	1.03 br s	1.08 s	1.19 s	1.32 s
21	2.25 s	2.38 s	2.34 s	2.32 s

Table 13-1-6: ¹H NMR spectroscopic data of pregnane-type steroids **13-1-12~13-1-16**.

H	13-1-12	13-1-13	13-1-14	13-1-15	13-1-16
1	1.68 m, 1.45 m	1.80 m, 1.57 m	1.85 m, 1.50 m	2.42 m, 1.57 m	2.35 m, 1.58 m
2	1.80 m	1.85 m, 1.52 m	1.85 m, 1.50 m	1.80 m	1.75 m
3	5.05 m	3.55 m	3.55 m	5.05 m	4.95 m
4	2.55 dd(15.3, 1.5) 2.25 dd(15.3, 1.5)	2.30 m	2.30 m	2.57 dd(15.1, 1.6) 2.27 dd(15.1, 1.6)	2.50 dd 2.25 dd
6	5.55 dd(5.55, 1.8)	5.55 dd(5.2, 1.6)	5.35 m	5.60 m	5.6 dd
7	3.84 brs	5.0 m	2.0 m, 1.55 m	3.8 br s	4.92 m
8	1.50 m	1.13 m	1.10 m	1.45 m	1.65 m
9	1.35 m	1.37 m	0.97 m	2.02 m	2.0 m
11	1.60 m, 1.45 m	1.60 m, 1.45 m	1.55 m, 1.45 m	4.1 br s	4.10 br s
12	1.72 m, 1.1 m	1.85 m, 1.70 m	1.73 m, 1.10 m	1.57 m, 1.1 m	1.43 m, 1.15 m
14	1.50 m	1.35 m	1.03 m	1.45 m	1.58 m
15	1.85 m, 1.25 m	1.57 m, 1.20 m	1.73 m, 1.23 m	1.85 m, 1.23 m	1.85 m, 1.62 m
16	1.60 m	1.80 m	1.55 m	1.9 m, 1.60 m	1.62 m, 1.20 m
17	2.03 m	2.01 m	2.0 m	2.06 m	2.10 m
18	0.60 s	0.60 s	0.6 s	0.65 s	0.60 s
19	1.00 s	1.00 s	1.0 s	1.25 s	1.17 s
20	5.77 m	5.75 m	5.75 m	5.75 m	5.73 m
21	5.0 m	5.0 m	5.0 m	5.0 m	5.0 m
OAc	2.0 s	2.0 s		2.05	2.06 s, 1.95 s

Table 13-1-7: ¹H NMR spectroscopic data of pregnane-type steroids **13-1-17~13-1-20**.

H	13-1-17	13-1-18	13-1-19	13-1-20
1	1.50 m, 1.90 m	2.56 m, 1.60 m	1.45 m, 0.98 m	α 1.25 m, β 1.75 m
2	1.80 m, 1.70 m	1.70 m	1.23 m, 1.09 m	3.28 ddd(11, 3, 3)
3	5.07 m	5.03 m	4.06 t(2.6)	4.13 br s
4	2.30 d(15.4), 2.60 d(15.2)	2.30 d(15.0), 2.60 d(14.9)	1.05 m, 1.25 m	α 1.65 m, β 1.40 m
5			1.65 m	0.97 m
6	5.80 m, 5.00 m	5.85 d(4.4)	1.21 m, 1.04 m	α 1.91 m, β 1.32 m
7	3.80 br s	4.95 m	1.44(obsc), 1.02 m	α 1.57 m, β 1.65 m
8	1.85 m	1.80 m	1.56 m	1.45 m
9	1.42 m	1.87 m	0.91 m	1.60 m
11	1.67 m, 1.50 m	4.13 br s	1.70 m, 1.45 m	α 1.65 m, β 1.35 m
12	1.10 m, 1.75 m	2.0 m, 1.18 m	1.40(obsc), 1.25 m	α 1.15 m, β 1.35 m
14	1.45 m	1.63 m	1.42 m	1.42 m
15	1.27 m, 1.87 m	1.20 m, 1.60 m	1.96 dd(14.5, 17.0) 2.20 dd(7.4, 17.0)	α 2.20 dd(7, 18) β 1.70 m
16	1.90 m, 1.60 m	1.85 m, 1.63 m		
17	2.0 m	2.10 m		1.65 m
18	0.65 s	0.65 s	1.01 s	0.68 s

Table 13-1-7 (continued)

H	13-1-17	13-1-18	13-1-19	13-1-20
19	4.55 d(11.9) 3.95 d(11.9)	4.85 dd(11.9) 4.22 dd(11.9)	0.83 s	0.82 s
20	5.75 m	5.75 m	6.49 q(7.5)	1.25 m, 1.60 m
21	5.00 m	5.00 m	1.85 d(7.5)	1.02 t(7.0)
OAc	2.10 s(6H)	2.05 s, 2.02 s, 1.95 s		
OMe				3.38 s

Table 13-1-8: ¹H NMR spectroscopic data of pregnane-type steroids **13-1-21~13-1-25**.

H	13-1-21	13-1-22	13-1-23	13-1-24	13-1-25
1	α 1.75 m, β 1.20 m	1.7 m, 2.00 m	1.23 m, 1.5 m	1.23 m, 1.50 m	1.72 m, 2.00 m
2	3.28 ddd(11, 3, 3)	2.32 m	1.60 m, 1.72 m	1.64 m, 1.71 m	2.4 m
3	4.11 br s		5.01 quin	5.00 quin	
4	α 1.63 m, β 1.40 m	5.73 d(1.7)	1.5 m	1.48 m, 1.56 m	5.07 s
5	0.86 m		1.5 m	1.47	
6	α 1.67 m, β 1.40 m	2.24 m, 2.24 m	1.22 m	1.20 m	2.34 dd, 2.40 m
7	α 1.07 m, β 1.58 m	1.01 m, 1.83 m	1.63 m	1.63 m, 1.60 m	1.1 m, 1.8 m
8	1.25 m	1.60 m	1.5 m	1.52 m	1.71 m
9	1.58 m	0.92 m	0.90 m	0.92 m	1.1 m
11	α 1.67 m, β 1.20 m	1.5 m, 1.62 m	1.31 m, 1.7 m	1.32 m, 1.63 m	1.67 m
12	α 1.08 m, β 1.37 m	1.20 m, 1.83 m	1.34 m, 1.9 m	1.34 m, 1.89 m	1.4 m, 2.00 m
14	1.40 m	0.90 m	1.4 m	1.44 m	1.41 m
15	α 2.75 dd(6, 10) β 2.08 dd(11, 18)	1.26 m 2.40 m	2.21 m	1.73 m 2.20 m	2.00 m 2.20 m
16		5.60 t(7.3)			
17	4.32 d(6)			1.64 m	2.0 m
18	0.83 s	0.94 s	0.83 s	0.67 s	0.81 m
19	0.77 s	1.2 s	1.00 s	0.81 s	1.2 s
20	5.78 ddd(6, 10, 16)	5.32 dq(1.6, 7.6)	6.58 q(7.3)	1.22 m, 1.63 m	5.10 q(6.1)
21	5.30 d(10) 5.37 d(16)	1.6 d(7.6)	1.83 d	1.00 t(7.3)	1.33 d
OAc		2.03 s	2.03 s	2.03 s	2.0 s
OMe	3.37 s ^①				

^① Typographic error exists in the original literature, uncorrectly printing the data of OMe (3.37) in the ¹³C NMR column.

13.1.3 Cardica glycosides

13.1.3.1 Furanone-type cardica glycosides

Table 13-1-9: Compounds, MFs, and test solvents of furanone-type cardica glycosides **13-1-26** and **13-1-27**.

No.	Compounds	MFs	Test solvents	References
13-1-26	6'-hydroxycalactin	C ₂₉ H ₄₀ O ₁₀	CD ₃ OD	[10]
13-1-27	6'-hydroxy-16 α -acetoxycalactin	C ₃₁ H ₄₂ O ₁₂	CD ₃ OD	[10]

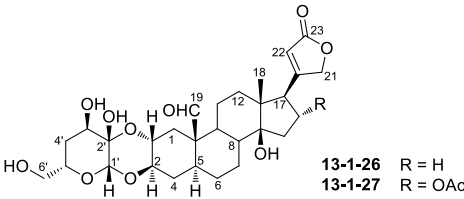


Table 13-1-10: ¹H NMR spectroscopic data of furanone-type cardica glycosides **13-1-26** and **13-1-27**.

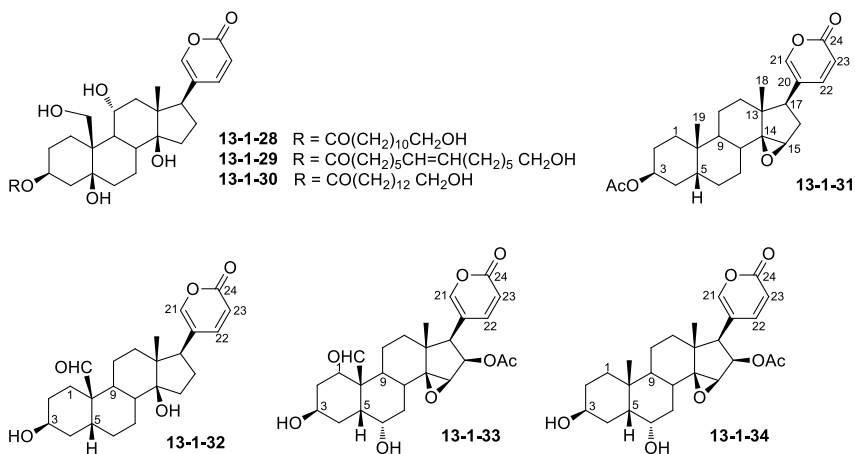
H	13-1-26	13-1-27	H	13-1-26	13-1-27
1	2.41 dd(12.5, 4.8) 1.13 t(12.5)	2.49 (12.5, 4.4) 1.19 (12.5)	16	2.19 m 1.90 m	5.92 dt(8.0, 4.0)
2	3.89 m	3.89 m	17	2.86 dd(5.7, 3.1)	2.74 d(4.0)
3	4.03 m	4.02 dt(10.9, 4.4)	18	0.85 s	0.83 s
4	1.67 m, 1.42 m	1.66 m, 1.40 m	19	10.07 s	10.07 s
5	1.59 m	1.59 m	21	5.05 dd(18.2, 1.6) 4.94 dd(18.2, 1.6)	4.98 br d(1.5)
6	2.27 m	2.25 m, 1.68 m	22	5.98 t(1.6)	5.98 br d(1.5)
7	2.05 m, 2.27 m	2.06 m	1'	4.75 s	4.50 s
8	1.62 m	1.62 m	3'	3.61 t(2.0)	3.61 t(2.0)
9	1.46 m	1.51 m	4'	1.85 m, 1.65 m	1.66 m
11	1.73 m, 1.31 m	1.80 m, 1.33 m	5'	3.70 m	3.65 m
12	1.53 m, 1.48 m	1.67 m	6'	3.60 m	3.61 m
15	2.12 m, 1.72 m	2.24 d(8.0)	OAc		2.06 s

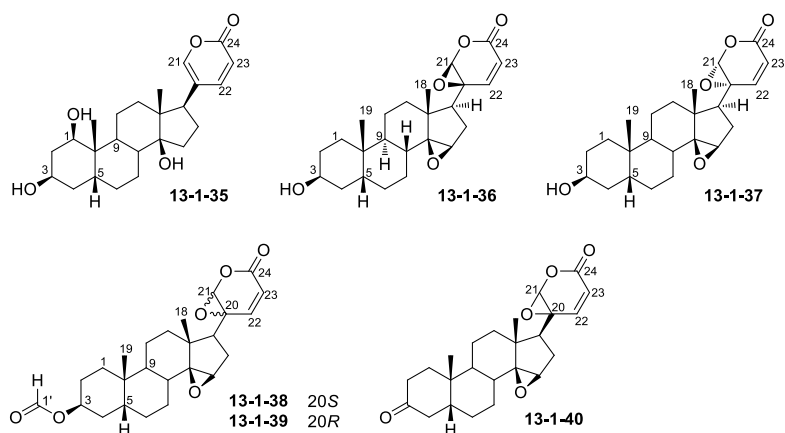
13.1.3.2 Pyranone-type cardica glycosides

Table 13-1-11: Compounds, MFs, and test solvents of pyranone-type cardica glycosides **13-1-28~13-1-40**.

No.	Compounds	MFs	Test solvents	Refer-ences
13-1-28	11 α ,19-dihydroxytelocinobufagin-3-(12-hydroxydodecanoic acid) ester	C ₃₆ H ₅₆ O ₉	CD ₃ OD	[11]
13-1-29	11 α ,19-dihydroxytelocinobufagin-3-(14-hydroxy-7-tetradecenoic acid) ester	C ₃₈ H ₅₈ O ₉	CD ₃ OD	[11]
13-1-30	11 α ,19-dihydroxytelocinobufagin-3-(14-hydroxytetradecanoic acid) ester	C ₃₈ H ₆₀ O ₉	CD ₃ OD	[11]
13-1-31	3 β -formyloxyresibufogenin	C ₂₅ H ₃₂ O ₅	CDCl ₃	[12]
13-1-32	19-oxobufalin	C ₂₄ H ₃₂ O ₅	CDCl ₃	[12]
13-1-33	19-oxodesacetylcinobufagin	C ₂₄ H ₃₀ O ₆	CD ₃ OD	[12]
13-1-34	6 α -hydroxycinobufagin	C ₂₆ H ₃₄ O ₇	CDCl ₃ - CD ₃ OD(7:1)	[12]
13-1-35	1 β -hydroxybufalin	C ₂₄ H ₃₄ O ₅ ^①	CDCl ₃	[12]
13-1-36	20S,21-epoxyresibufogenin	C ₂₄ H ₃₂ O ₅	—	[13]
13-1-37	20R,21-epoxyresibufogenin	C ₂₄ H ₃₂ O ₅	—	[13]
13-1-38	3-O-formyl-20S,21-epoxyresibufogenin	C ₂₅ H ₃₂ O ₆	—	[13]
13-1-39	3-O-formyl-20R,21-epoxyresibufogenin	C ₂₅ H ₃₂ O ₆	—	[13]
13-1-40	3-oxo-20S,21-epoxyresibufogenin	C ₂₄ H ₃₀ O ₅	—	[13]

^① The molecular formula in the experimental section was incorrect, while that in the main body was right.



**Table 13-1-12:** ^1H NMR spectroscopic data of pyranone-type cardica glycosides 13-1-28~13-1-32.

H	13-1-28	13-1-29	13-1-30	13-1-31	13-1-32
1	2.25 dd(15.7, 4.0) 2.19 m	2.18 m, 2.25 m	2.18 m, 2.22 m	α 1.60 m, β 1.41 m	α 1.54 m, β 1.81 m
2	1.68 m, 2.01 m	1.67 m, 2.01 m	1.67 m, 2.01 m	α 1.59 m, β 1.68 m	α 1.54 m, β 1.71 m
3	5.11 br s ($W_{1/2} = 11$)	5.11 br s ($W_{1/2} = 12$)	5.11 br s ($W_{1/2} = 11$)	α 5.25 br s	α 4.19 brt(2.3)
4	2.25 br dd(15.7, 4.0) 1.54 m	2.25 br dd(15.7, 4.0) 1.54 m	1.54 m, 2.25 m	α 1.90 m	α 1.87 qd(14.2, 2.8) β 1.53 m
5				β 1.48 m β 1.72 m	β 2.33 m
6	1.87 brtd(13.5, 4.4) 1.51 m	1.51 m, 1.88 m	1.51 m, 1.88 m	α 1.23 m	α 1.44 m
7	1.27 m, 2.03 m	1.30 m, 2.03 m	1.30 m, 2.03 m	β 1.85 dt(13.8, 4.6) α 0.94 qd(12.8, 3.7) β 1.53 m	β 1.60 m α 1.27 m β 1.81 m
8	1.86 m	1.86 m	1.86 m	β 2.01 td(11.9, 3.7)	β 1.83 m
9	1.75 m	1.75 m	1.76 m	α 1.56 m	α 1.69 m
11	3.38 td(10.4, 4.0)	3.38 br td(10.5, 4.0)	3.88 brtd(10.1, 3.2)	α 1.53 m	α 1.47 m
12	1.66 br dd(13.3, 4.0) 1.54 m	1.67 m 1.53 m	1.66 m 1.53 m	β 1.33 m α 1.40 m β 1.66 m	β 1.81 m α 1.35 td(13.3, 3.7) β 1.55 m

Table 13-1-12 (continued)

H	13-1-28	13-1-29	13-1-30	13-1-31	13-1-32
15	2.14 m	2.12 m	2.14 m	α 3.53 s	α 2.05 dt(12.8, 9.6)
	1.74 m	1.73 m	1.74 m		β 1.67 m
16	1.77 m, 2.20 m	1.77 m, 2.21 m	1.76 m, 2.19 m	α 2.39 ddd (15.1, 10.5, 1.4) β 1.96 d(15.1)	α 2.20 dt(12.8, 9.6) β 1.76 m
17	2.62 dd(9.2, 6.5)	2.62 dd(9.3, 6.5)	2.62 br dd(9.5, 6.7)	α 2.47 d(10.1)	α 2.47 dd(9.6, 6.4)
18	0.75 s	0.75 s	0.75 s	0.78 s	0.78 s
19	4.22 d(11.3) 3.84 d(11.3)	4.21 d(11.1) 3.84 d(11.1)	4.20 d(10.9) 3.84 d(11.3)	1.01 s	9.50 d(1.4)
21	7.44 dd(2.4, 0.8)	7.44 br dd(2.4, 0.8)	7.44 br d(2.0)	7.24 d(1.8)	7.24 d(2.3)
22	7.94 dd(9.7, 2.4)	7.94 dd(9.7, 2.4)	7.94 dd(9.7, 2.4)	7.79 dd(9.6, 2.3)	7.84 dd(9.6, 2.3)
23	6.27 dd(9.7, 0.8)	6.27 dd(9.7, 0.8)	6.27 d(9.7)	6.25 dd(9.6, 0.9) 2.07 s	6.27 d(9.6)
OAc					
2'	2.33 td(7.3, 4.8)	2.34 td(7.3, 4.0)	2.33 td(7.3, 4.4)		
3'	1.62 t(7.3)	1.63 m	1.63 m		
4'	1.32 m	1.34 m	1.33 m		
5'	1.31 m	1.37 m	1.29 m		
6'	1.31 m	2.05 m	1.29 m		
7'	1.31 m	5.36 m	1.29 m		
8'	1.31 m	5.36 m	1.29 m		
9'	1.31 m	2.05 m	1.29 m		
10'	1.31 m	1.37 m	1.29 m		
11'	1.52 m	1.35 m	1.29 m		
12'	3.54 t(6.9)	1.35 m	1.34 m		
13'		1.54 m	1.53 m		
14'		3.54 td(6.4, 4.0)	3.53 t(6.5)		

Table 13-1-13: ^1H NMR spectroscopic data of pyranone-type cardica glycosides 13-1-33~13-1-36.

H	13-1-33	13-1-34	13-1-35	13-1-36
1	α 1.53 m, β 1.85 m	α 1.54 m, β 1.48 m	α 3.84 s	1.51 (2H)
2	1.63 (2H)	1.55 (2H)	α 1.76 m, β 2.00 m	1.53 (2H)
3 α	4.08 br t(2.3)	4.15 s	4.20 m	4.13 br t(2.3)
4	1.95 td(13.8, 2.8)	1.73 m	1.96 m	1.88 td(13.8, 2.8)
4 β	1.48 m	1.51 m	1.48 m	1.34
5 β	2.30 m	1.96 dt(13.3, 4.1)	2.10 m	1.77

Table 13-1-13 (continued)

H	13-1-33	13-1-34	13-1-35	13-1-36
6	α 1.36 m, β 1.54 m	β 4.09 dt(11.9, 4.6)	α 1.39 m, β 1.85 m	α 1.21, β 1.84
7 α	1.05 qd(12.8, 3.2)	0.95 m	1.32 m	0.95 qd(12.8, 3.7)
7 β	1.48 m	1.71 m	1.76 m	1.47
8 β	2.28 m	2.12 td(12.4, 3.7)	1.60 td(11.9, 3.7)	1.99 td(11.9, 3.7)
9 α	1.82 m	1.59 m	1.47 m	1.57
11 α	1.59 m	1.56 m	1.32 m	1.50
11 β	1.91 td(12.8, 3.7)	1.28 qd(13.3, 3.2)	1.24 td(13.7, 3.7)	1.30
12 α	1.46 m	1.44 td(13.3, 3.2)	1.29 m	1.32
12 β	1.77 m	1.80 dt(13.3, 2.8)	1.51 m	1.60
15	α 3.59 s	α 3.68 s	α 2.00 m, β 1.76 m	α 3.56 s
16	α 4.72 dd(9.2, 1.4)	α 5.47 d(9.2)	α 2.19 m, β 1.71 m	2.27(2H)
17	α 2.69 d(9.2)	α 2.85 d(9.2)	α 2.46 dd(9.6, 6.4)	α 1.46
18	0.85 s	0.82 s	0.71 s	1.02 s
19	9.47 d(1.8)	0.97 s	1.10 s	0.99 s
21	7.40 s	7.22 br s	7.23 d(1.8)	5.30 s
22	8.10 d(9.6)	7.96 br s	7.84 dd(9.6, 2.3)	7.93 d(10.5)
23	6.20 dd(9.6, 0.9)	6.24 d(10.2)	6.23 d(9.6)	6.03 d(10.5)
2'		1.91 s		

Table 13-1-14: ¹H NMR spectroscopic data of pyranone-type cardica glycosides 13-1-37~13-1-40.

H	13-1-37	13-1-38	13-1-39	13-1-40
1	1.51 (2H)	α 1.58, β 1.38	α 1.59, β 1.37	α 2.04, β 1.47
2	1.54 (2H)	α 1.23, β 1.85	α 1.21, β 1.85	α 2.32, β 2.21
3	α 4.13 brt(2.3)	α 5.24 br s	α 5.23 s	
4	α 1.89, β 1.35	α 1.89, β 1.48	α 1.87, β 1.46	α 2.60, β 2.04
5	β 1.76	β 1.70	β 1.70	β 1.85
6	α 1.21, β 1.86	α 1.53, β 1.67	α 1.52, β 1.67	α 1.31, β 1.85
7	α 0.93 qd(13.3, 3.7)	α 0.93 qd(13.3, 4.1)	α 0.91 qd(13.3, 4.1)	α 1.00 qd(12.8, 3.2)
	β 1.47	β 1.47	β 1.47	β 1.60
8	β 2.05 td(11.9, 3.7)	β 2.00 td(11.9, 3.7)	β 2.04	β 2.06
9	α 1.57 td(11.5, 3.7)	α 1.53	α 1.54	α 1.73 td(11.5, 3.2)
11	α 1.49, β 1.32	α 1.51, β 1.30	α 1.50, β 1.33	α 1.60, β 1.42
12	α 1.34	α 1.30	α 1.33	α 1.38
	β 1.70 dd(9.6, 2.8)	β 1.60	β 1.69	β 1.67
15	α 3.45 s	α 3.57 s	3.45 s	3.59 s
16	α 2.08, β 1.89	2.29 (2H)	α 2.10 s, β 1.89 s	2.30 (2H)
17	α 1.77	α 1.45	1.77	1.50
18	1.18 s	1.03 s	1.19 s	1.05 s
19	1.00 s	1.00 s	1.02 s	1.05 s
21	5.40 s	5.30 s	5.40 s	5.31 d(0.9)
22	7.69 br d(7.3)	7.93 dd(10.5, 0.9)	7.69 br d(7.3)	7.93 dd(10.5, 0.9)
23	6.06 d(10.1)	6.04 d(10.1)	6.06 d(10.1)	6.05 d(10.1)
1'		8.07 s	8.07 s	

13.1.4 Cholestane-type steroids

Table 13-1-15: Compounds, MFs, and test solvents of cholestane-type steroids **13-1-41**~**13-1-44**.

No.	Compounds	MFs	Test solvents	References
13-1-41	hyousterone A	C ₂₇ H ₄₂ O ₅	DMSO- <i>d</i> ₆	[14]
13-1-42	hyousterone B	C ₂₇ H ₄₂ O ₅	DMSO- <i>d</i> ₆	[14]
13-1-43	hyousterone C	C ₂₇ H ₄₂ O ₆	DMSO- <i>d</i> ₆	[14]
13-1-44	hyousterone D	C ₂₇ H ₄₂ O ₆	DMSO- <i>d</i> ₆	[14]

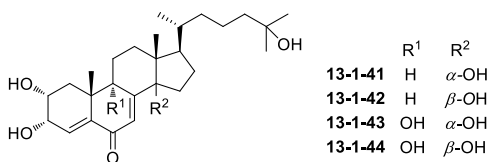


Table 13-1-16: ¹H NMR spectroscopic data of cholestane-type steroids **13-1-41**~**13-1-44**.

H	13-1-41	13-1-42	13-1-43	13-1-44
1	α 1.76 m, β 1.56 m	α 1.73 m, β 1.60 m	α 2.37 t(12.0), β 1.28 m	α 2.29 m, β 1.27 m
2	3.63 m	3.63 m	3.69 m	3.68 m
3	3.99 m	3.99 m	4.02 br s	
4	6.27 d(5.3)	6.29 d(5.2)	6.46 d(5.2)	6.50 d(5.1)
7	5.82 s	6.30 s	5.90 s	6.33 s
9	2.82 dd(7.7, 11.4)	2.56 dd(6.9, 10.0)		
11	α 1.66 m, β 1.55 m	α 1.66 m, β 1.51 m	α 1.61 m, β 2.01 m	α 1.43 m, β 1.84 m
12	α 2.02 m, β 1.63 m	α 1.90 m, β 1.33 m	α 1.24 m, β 1.24 m	α 1.75 m, β 1.29 m
15	α 1.79 m, β 1.58 m	α 1.50 m, β 1.44 m	α 1.66 m, β 1.77 m	α 2.23 m, β 1.36 m
16	α 1.88 m, β 1.28 m	α 1.68 m, β 1.68 m	α 1.89 m, β 1.28 m	α 0.89 m, β 1.63 m
17	1.81 m	1.50 m	1.83 m	1.49 m
18	0.63 s	0.91 s	0.66 s	0.91 s
19	1.03 s	0.98 s	1.14 s	1.11 s
20	1.40 m	1.50 m	1.40 m	1.48 m
21	0.90 d(6.7)	0.88 d(5.9)	0.91 d(6.5)	0.88 d(5.7)
22	1.34 m, 1.02 m	1.61 m, 0.92 m	1.33 m, 1.02 m	1.63 m, 1.63 m
23	1.37 m, 1.19 m	1.36 m, 1.17 m	1.89 m, 1.35 m	1.23 m, 1.23 m
24	1.34 m, 1.25 m	1.34 m, 1.26 m	1.34 m, 1.24 m	1.34 m, 1.25 m
26	1.07 s	1.06 s	1.06 s	1.06 s
27	1.07 s	1.06 s	1.06 s	1.06 s
2-OH	4.41	4.42	4.58	4.56
3-OH	4.76	4.74	4.67	4.62
9-OH			5.33	4.83
14-OH	4.70	4.67	5.37	4.62
25-OH	4.05	4.08	4.09	4.04

13.1.5 Ergostane-type steroids

Table 13-1-17: Compounds, MFs, and test solvents of ergostane-type steroids 13-1-45~13-1-53.

No.	Compounds	MFs	Test solvents	Refer- ences
13-1-45	3 β -hydroxy-4 α ,14 α -dimethyl-5 α -ergosta-8,24(28)-dien-11-one	C ₃₀ H ₄₈ O ₂	CDCl ₃	[15]
13-1-46	3 β ,11 α -dihydroxy-4 α ,14 α -dimethyl-5 α -ergosta-8,24(28)-dien-7-one	C ₃₀ H ₄₈ O ₃	CDCl ₃	[15]
13-1-47	3 β ,7 α -dihydroxy-4 α ,14 α -dimethyl-5 α -ergosta-8,24(28)-dien-11-one	C ₃₀ H ₄₈ O ₃	CDCl ₃	[15]
13-1-48	(20 <i>S</i> ,22 <i>R</i> ,24 <i>R</i>)-16,22-epoxy-3 β ,14 α ,23 β ,25-tetrahydroxyergost-7-en-6-one	C ₂₈ H ₄₄ O ₆	C ₅ D ₅ N	[16]
13-1-49	(23 <i>R</i> ,24 <i>R</i> ,25 <i>R</i>)-23,26-epoxy-3 β ,14 α ,21 α ,22 α -tetrahydroxyergost-7-en-6-one	C ₂₈ H ₄₄ O ₆	C ₅ D ₅ N	[16]
13-1-50	22,23-epoxy-3 β ,14 α ,20 β ,24 β -tetrahydroxy-7-en-6-one	C ₂₈ H ₄₄ O ₆	C ₅ D ₅ N	[16]
13-1-51	7-nor-ergosterolide	C ₂₇ H ₄₂ O ₃	CDCl ₃	[17]
13-1-52	3 β ,11 α -dihydroxyergosta-8,24(28)-dien-7-one	C ₂₈ H ₄₄ O ₃	CDCl ₃	[17]
13-1-53	3 β -hydroxyergosta-8,24(28)-dien-7-one	C ₂₈ H ₄₄ O ₂	CDCl ₃	[17]

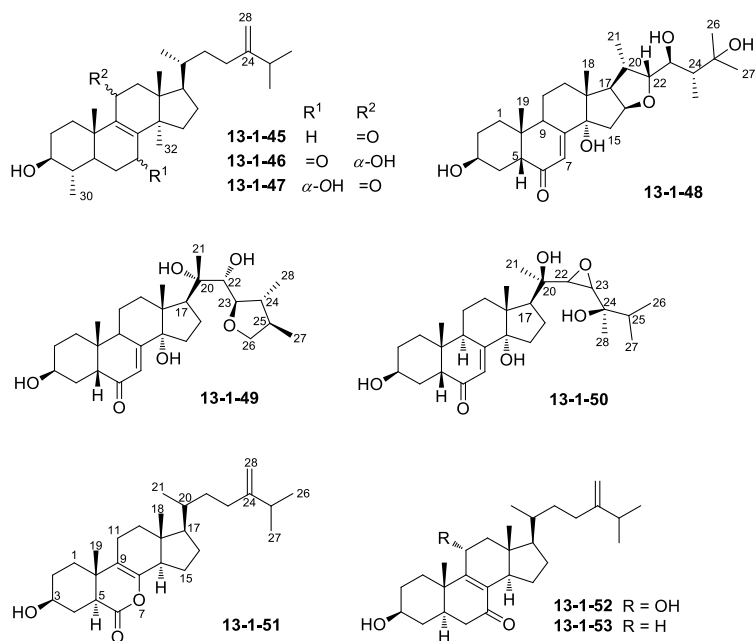


Table 13-1-18: ¹H NMR spectroscopic data of ergostane-type steroids **13-1-45~13-1-47**.

H	13-1-45	13-1-46	13-1-47
1	α 1.02 m, β 2.97 dt(13.5, 3.8)	α 1.90 m, β 1.93 dt(13.2, 3.3)	α 1.10 m, β 2.98 dt(13.5, 3.9)
2	α 1.84 m, β 1.58 m	α 1.96 m, β 1.62 m	α 1.86 m, β 1.58 m
3	α 3.11 ddd(11.0, 9.5, 5.0)	α 3.19 ddd(11.1, 9.8, 4.8)	α 3.17 ddd(11.0, 9.8, 5.2)
4 β	1.42 m	1.47 m	1.45 m
5 α	0.86 m	1.76 ddd(11.6, 13.6, 3.5)	1.20 ddd(15.6, 13.3, 2.1)
6 α	1.80 m	2.56 dd(16.0, 3.5)	1.48 m
6 β	1.24 m	2.24 dd(16.0, 14.0)	1.72 m
7	α 2.28 m, β 2.31 ddd(20.3, 7.0, 2.0)		β 4.35 dd(4.5, 2.2)
11		β 4.54 dd(9.5, 5.4)	
12 α	2.67 dd(16.5, 1.0)	2.50 dd(13.5, 9.5)	2.71 dd(17.2, 0.9)
12 β	2.50 d(16.5)	1.86 dd(13.5, 5.4)	2.50 d(17.2)
15 α	1.35 m	2.07 m	1.70 ddd(11.7, 9.4, 1.8)
15 β	1.77 m	1.65 m	1.89 m
16 α	2.05 m	1.99 m	2.05 m
16 β	1.38 m	1.30 m	1.43 m
17 α	1.75 m	1.58 m	1.78 m
18	0.84 s	β 0.67 s	β 0.81 s
19	1.11 s	1.24 s	1.04 s
20	1.42 m	1.36 m	1.42 m
21	0.91 d(6.4)	0.94 d(6.4)	0.91 d(6.4)
22	1.16 m, 1.45 m	1.14 m, 1.57 m	1.16 m, 1.58 m
23	1.89 m	1.89 m	1.88 m
	2.12 ddd(15.0, 11.5, 5.0)	2.13 m	2.12 m
25	2.23 sept(6.9)	2.23 sept(6.9)	2.23 sept(6.9)
26	1.03 d(6.9)	1.02 d(6.9)	1.03 q(6.9)
27	1.03 d(6.9)	1.03 d(6.9)	1.03 q(6.9)
28	4.54 d(1.5), 4.55 s	4.66 d(1.4), 4.72 s	4.67 d(1.4), 4.73 s
30	1.02 s	1.01 q(6.4)	1.04 d(6.2)
32	1.14 s	1.13 q	1.27 s

Table 13-1-19: ¹H NMR spectroscopic data of ergostane-type steroids **13-1-48~13-1-50**.

H	13-1-48	13-1-49	13-1-50	H	13-1-48	13-1-49	13-1-50
3	4.13 br s	4.12 br s	4.15 br s	21	1.39 d(7.1)	1.70 s	1.59 s
5	2.97 m	2.98 m	2.97 m	22	4.44 dd(5.6, 9.2)	3.89 m	3.64 d(1.9)
7	6.18 br s	6.26 br s	6.21 br s	23	4.69 br d(9.1)	3.89 m	3.31 d(1.9)
9	3.54 br s	3.56 br s	3.54 br s	24	2.21 m	1.94 m	1.92 m
16	5.29 m	2.47 m	2.43 m	25		1.70 m	1.92 m
		2.30 m	2.33 m				
17	2.85 d(8.0)	3.46 t(9.1)	3.05 t(9.3)	26	1.47 s	3.89 m	1.16 d(6.8)
						3.38 t(8.4)	
18	0.97 s	1.23 s	1.14 s	27	1.56 s	0.87 d(6.6)	1.16 d(6.8)
19	1.04 s	1.05 s	1.04 s	28	1.45 d(7.0)	1.27 d(6.5)	1.38 s
20	2.70 m						

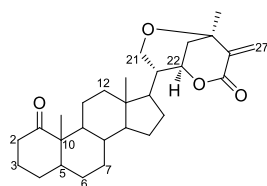
Table 13-1-20: ^1H NMR spectroscopic data of ergostane-type steroids **13-1-51~13-1-53**.

H	13-1-51	13-1-52	13-1-53
1	1.70 m, 1.44 m	1.93 m, 1.78 m	1.82 m, 1.34 m
2	1.51 m, 1.39 m	1.55 m, 1.46 m	1.52 m, 1.38 m
3	3.63 m	3.67 m	3.62 m
4	1.86 m, 1.51 m	1.87 m, 1.70 m	1.66 m, 1.43 m
5	2.38 dd(3.3, 12.7)	2.06 m	1.93 m
6		2.43 m, 2.23 dd(3.8, 16.2)	2.38 m, 2.15 dd(3.8, 16.2)
11	2.20 m, 2.14 m	4.57 dd(4.6, 7.8)	2.41 m, 2.28 m
12	2.04 m, 1.43 m	2.42 m, 1.66 dd(4.6, 14.0)	2.03 m, 1.39 m
14	2.31 m	2.41 m	2.10 m
15	2.04 m, 1.45 m	1.96 m, 1.32 m	1.97 m, 1.33 m
16	1.72 m, 1.44 m	2.32 m, 1.57 m	2.41 m, 1.31 m
17	1.25 m	1.32 m	1.14 m
18	0.67 s	0.56 s	0.58 s
19	0.96 s	1.18 s	1.16 s
20	1.42 m	1.39 m	1.39 m
21	0.95d(6.5)	0.96 d(6.3)	0.95 d(6.5)
22	1.54 m, 1.14 m	1.56 m, 1.14 m	1.54 m, 1.14 m
23	2.08 m	2.07 m	2.06 m
25	2.21 m	2.22 m	2.20 m
26	1.02 d(6.8)	1.03 d(6.9)	1.01 d(6.8)
27	1.01 d(6.8)	1.01 d(6.9)	1.00 d(6.8)
28	4.71 s, 4.65 s	4.71 s, 4.65 s	4.70 s, 4.64 s

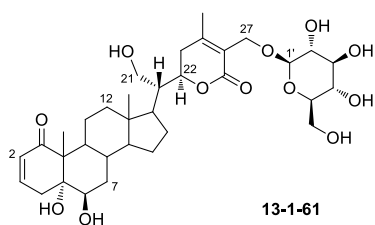
13.1.6 Ergoline-type steroids

Table 13-1-21: Compounds, MFs, and test solvents of ergoline-type steroids **13-1-54~13-1-63**.

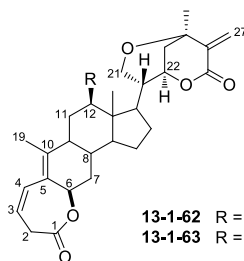
No.	Compounds	MFs	Test solvents	References
13-1-54	withametelin I	$\text{C}_{28}\text{H}_{36}\text{O}_6$	CDCl_3	[18]
13-1-55	withametelin J	$\text{C}_{28}\text{H}_{38}\text{O}_7$	CDCl_3	[18]
13-1-56	withametelin K	$\text{C}_{28}\text{H}_{36}\text{O}_6$	CDCl_3	[18]
13-1-57	withametelin L	$\text{C}_{28}\text{H}_{36}\text{O}_5$	CDCl_3	[18]
13-1-58	withametelin M	$\text{C}_{28}\text{H}_{36}\text{O}_5$	$\text{CDCl}_3\text{-CD}_3\text{OD}$	[18]
13-1-59	withametelin N	$\text{C}_{28}\text{H}_{38}\text{O}_6$	CDCl_3	[18]
13-1-60	withametelin O	$\text{C}_{28}\text{H}_{38}\text{O}_6$	CDCl_3	[18]
13-1-61	withametelin P	$\text{C}_{34}\text{H}_{50}\text{O}_{12}$	$\text{CDCl}_3\text{-CD}_3\text{OD}$	[18]
13-1-62	1,10- <i>seco</i> -withametelin B	$\text{C}_{28}\text{H}_{36}\text{O}_5$	CDCl_3	[18]
13-1-63	12 β -hydroxy-1,10- <i>seco</i> -withametelin B	$\text{C}_{28}\text{H}_{36}\text{O}_6$	CDCl_3	[18]



- 13-1-54** 5 β ,6 β -epoxy, 12 β -OH, Δ^2
13-1-55 5 α -OH, 6 β -OH, 12 β -OH, Δ^2
13-1-56 6 β -OH, 12 β -OH, $\Delta^{2,4}$
13-1-57 12 β -OH, $\Delta^{2,5}$
13-1-58 12 β -OH, $\Delta^{3,5}$
13-1-59 3 α -OH, 5 β ,6 β -epoxy
13-1-60 5 β -OH, 6 α -OH, Δ^2



13-1-61



- 13-1-62** R = H
13-1-63 R = OH

Table 13-1-22: ^1H NMR spectroscopic data of ergoline-type steroids **13-1-54**~**13-1-56**.

H	13-1-54	13-1-55	13-1-56
2	6.02 dd(10, 2.5)	5.87 dd(10.2, 2.4)	5.96 dd(9.6, 0.9)
3	6.85 ddd(10, 6, 2.4)	6.59 ddd(10.2, 4.8, 2.4)	6.99 dd(9.6, 6)
4	3.01 dd(21.6, 2.4), 1.90 m	3.34 dt(19.8, 2.7), 2.07 m	6.15 d(6)
6	3.13 d(2.4)	3.65 br s(ov)	4.40 br s
7	2.10 dd(14, 4.3), 1.18 m	1.58~1.68 m	2.01 m, 1.18 m
8	1.48 m	1.66 m	1.98 m
9	1.18 m	1.90 m	1.90 m
11	2.20 dt(12.9, 4.2), 1.45 m	2.43 dt(12.3, 3.3), 1.34 m	2.02 m, 1.55 m
12	3.52 dd(11.1, 4.2)	3.65 (ov)	3.36 dd(11, 4.8)
14	0.98 m	1.24 m	1.15 m
15	1.65 m	1.24~1.70 m	1.70 m
16	1.78 m	1.75 m	1.70 m
17	1.80 m	1.88 m	1.95 m
18	0.70 s	0.70 s	0.74 s
19	1.24 s	1.30 s	1.45 s
20	1.85 m	1.90 m	1.85 m
21	4.54 d(13.5)	4.54 d(13.5)	4.56 d(13.5)
	3.70 dd(13.5, 3.3)	3.68 dd(13.5, 2.7)	3.62 dd(13.5, 3)
22	4.66 br s	4.69 br s	4.69 br s
23	1.85~1.95 m	1.58~1.68 m	1.98 m
27	6.75 s, 5.99 s	6.75 s, 6.00 s	6.96 s, 6.01 s
28	1.42 s	1.42 s	1.42 s

Table 13-1-23: ¹H NMR spectroscopic data of ergoline-type steroids **13-1-57~13-1-61**.

H	13-1-57	13-1-58	13-1-59	13-1-60	13-1-61
2	5.85 dd(9.9, 2.1)	3.28 d(20.4)	2.78 dd(14.1, 6.3)	6.02 dd(9.9, 2.4)	5.77 dd(9.9, 2.4)
		2.75 dd(20.4, 4.5)	2.64 dd(14.1, 5.4)		
3	6.79 ddd(9.9, 5.1, 2.7)	5.60 dt(9.6, 4.5)	4.32 br m	6.75 ddd(9.9, 5.4, 2.4)	6.64 ddd(9.9, 9.5, 2.1)
4	3.27 dd(21.3, 2.1)	6.05 dd(9.6, 2.1)	2.33 dd(14.7, 4.5)	2.80 dt(20.7, 2.7)	3.25 dt(19.8, 2.7)
	2.85 dd(21.3, 4.8)		1.45 m	2.50 dd(20.7, 5.1)	2.05 dd(19.8, 5.1)
6	5.55 d(6)	5.64 d(5.1)	3.24 d(1.5)	3.88 dd(11.7, 5.1)	3.52 br s
7	1.98 m	2.18 dt(17.7, 5.1)	2.12 dt(14.1, 2.7)	1.98 m	1.70 m
	1.51 m	1.52 m	1.44 m		1.50 m
8	1.36 m	1.38 m	1.48 m	1.20 m	1.78 m
9	1.74 m	1.89 m	1.20 m	1.30 m	1.80 m
11	2.40 dt(12.6, 4.2)	1.98 m	1.35~1.45 m	1.36 m	2.22 m
	1.50 m	1.35 m			
12	3.71 (ov)	3.71 (ov)	1.40 m	1.80~1.85 m 1.25~1.30 m	2.05 m 1.45 m
14	1.13 m	1.13 m	0.98 m	1.04 m	1.24 m
15	1.28~1.70 m	1.70 m	1.65 m	1.70 m	1.70 m, 1.24 m
16	1.46~1.78 m	1.42 m	1.70 m	1.73 m	1.82 m
17	1.88 m	1.86 m	1.78 m	1.68~1.80 m	1.78 m
18	0.70 s	0.72 s	0.62 s	0.65 s	0.80 s
19	1.21 s	1.35 s	1.14 s	1.15 s	1.30 s
20	1.88 m	1.85 m	1.80 m	1.75~1.85 m	1.90 m
21	4.58 d(13.5)	4.56 d(13.5)	3.83 d(13.2)	3.82 d(13.2)	3.78 dd(11.4, 4.2)
	3.68 dd(13.5, 3)	3.68 dd(13.5, 3.0)	3.67 dd(13.2, 3.3)	3.67 dd(13.2, 2.7)	3.92 dd(11.4, 2.1)
22	4.69 br s	4.69 br s	4.61 br s	4.61 br s	4.56 dt(13.2, 3.3)
23	1.92~2.04 m	1.91~1.98 m	1.80~1.97 m	1.85~2.00 m	2.97 d(18.6)
					2.32 dd(18.6, 2.7)
27	6.75 s	6.76 s	6.71 s	6.73 s	4.62 d(11.4)
	6.00 s	6.01 s	5.98 s	5.99 s	4.47 d(11.4)
28	1.43 s	1.43 s	1.37 s	1.39 s	1.42 s
1'					4.32 d(7.8)
2'					3.16 dd(8.7, 7.8)
3'					3.26 t(8.7)
4'					3.28 t(8.7)
5'					3.32 dt(9.5, 2.4)
6'					3.86 d(12)
					3.66 dd(12, 4.8)

Table 13-1-24: ^1H NMR spectroscopic data of ergoline-type steroids **13-1-62** and **13-1-63**.

H	13-1-62	13-1-63	H	13-1-62	13-1-63
2	3.79 br d(17.4) 3.18 dd(17.4, 8.7)	3.82 br d(17.4) 3.18 dd(17.4, 8.7)	16	1.80 m	1.80 m
3	5.50 dd(11.4, 8.7)	5.50 dd(11.4, 8.7)	17	1.80 m	1.90 m
4	6.55 dd(11.4, 3.3)	6.55 dd(11.4, 3.3)	18	0.71 s	0.72 s
6	5.1 br d(3.3)	5.10 br d(3.3)	19	1.79 s	1.79 s
7	2.01 m	2.12 dd(12.3, 4.2) 1.34 dd(12.3, 3.6)	20	1.90 m	1.90 m
8	1.69 m	1.40 m	21	3.88 d(13.5) 3.74 dd(13.5, 2.7)	4.50 d(13.5) 3.68 d(13.5)
9	1.69 m	1.69 m	22	4.66 br s	4.68 br s
11	1.25 m, 1.65 m	2.05 m, 1.38 m	23	1.85~2.0 m	1.85~2.05 m
12	2.05 m	3.60 dd(10.8, 4.5)	27	6.76 s, 6.01 s	6.75 s, 6.01 s
14	1.25 m	1.16 m	28	1.43 s	1.42 s
15	1.90 m	1.78 m			

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13.2 Some additional steroids

Table 13-2-1: Compounds, MFs, and test solvents of steroids 13-2-1~13-2-50.

No.	Compounds	MFs	Test solvents	References
13-2-1	20- <i>epi</i> -3-dehydroxy-3-oxo-5,6-dihydro-4,5-dehydroverazine	C ₂₇ H ₄₁ NO	CDCl ₃	[19]
13-2-2	20- <i>epi</i> -verazine	C ₂₇ H ₄₃ NO	CDCl ₃	[19]
13-2-3	20- <i>epi</i> -4β-hydroxyverazine	C ₂₇ H ₄₃ NO ₂	CDCl ₃	[19]
13-2-4	20- <i>epi</i> -25β-hydroxyverazine	C ₂₇ H ₄₃ NO ₂	CDCl ₃	[19]
13-2-5	verazine	C ₂₇ H ₄₃ NO	CDCl ₃	[19]
13-2-6	4β-hydroxyverazine	C ₂₇ H ₄₃ NO ₂	CDCl ₃	[19]
13-2-7	25β-hydroxyverazine	C ₂₇ H ₄₃ NO ₂	CDCl ₃	[19]
13-2-8	20- <i>epi</i> -4β-hydroxyverazine triacetate ^①	C ₃₃ H ₄₉ NO ₅	CDCl ₃	[19]
13-2-9	20- <i>epi</i> -25β-hydroxyverazine diacetate ^①	C ₃₁ H ₄₇ NO ₄	CDCl ₃	[19]
13-2-10	ecliptalbine	C ₂₇ H ₃₉ NO ₂	CDCl ₃	[19]
13-2-11	4β-hydroxyverazine triacetate ^①	C ₃₃ H ₄₉ NO ₅	CDCl ₃	[19]
13-2-12	25β-hydroxyverazine diacetate	C ₃₁ H ₄₇ NO ₄	CDCl ₃	[19]
13-2-13	veramiline ^②	C ₂₇ H ₄₅ NO	CDCl ₃	[19]
13-2-14	salpichrolide L	C ₂₈ H ₃₆ O ₅	—	[20]
13-2-15	salpichrolide M	C ₂₈ H ₃₆ O ₆	—	[20]
13-2-16	salpichrolide M acetate ^①	C ₃₀ H ₃₈ O ₇	—	[20]
13-2-17	salpichrolide J	C ₂₈ H ₃₄ O ₅	—	[20]
13-2-18	salpichrolide K	C ₂₈ H ₃₆ O ₆	—	[20]
13-2-19 ^③	—	C ₂₈ H ₃₈ O ₇	—	[20]
13-2-20	mexrenone	C ₂₄ H ₃₂ O ₅	CDCl ₃	[21]
13-2-21	canrenone	C ₂₂ H ₂₈ O ₃	CDCl ₃	[21]
13-2-22	11α-hydroxymexrenone	C ₂₄ H ₃₂ O ₆	CDCl ₃	[21]
13-2-23	12β-hydroxymexrenone	C ₂₄ H ₃₂ O ₆	CDCl ₃	[21]
13-2-24	9α-hydroxycanrenone	C ₂₂ H ₂₈ O ₄	CDCl ₃	[21]
13-2-25	17β-hydroxypetuniasterone A-7-acetate	C ₃₄ H ₄₈ O ₈ S	CDCl ₃	[22]
13-2-26	—	C ₃₀ H ₄₄ O ₅	CDCl ₃	[22]
13-2-27	—	C ₃₄ H ₄₈ O ₈	CDCl ₃	[22]
13-2-28	—	C ₃₂ H ₄₆ O ₇	CDCl ₃	[22]
13-2-29	petuniasterone I	C ₃₄ H ₄₈ O ₉ S	CDCl ₃	[22]
13-2-30	petuniasterone J	C ₃₄ H ₄₈ O ₉	CDCl ₃	[22]
13-2-31	petuniasterone K	C ₃₂ H ₄₄ O ₇	CDCl ₃	[22]
13-2-32	petuniasterone L	C ₃₄ H ₄₆ O ₈ S	CDCl ₃	[22]
13-2-33	12ξ-acetoxy-11β-hydroxypetuniasterone D-7-acetate	C ₃₄ H ₄₈ O ₉	CDCl ₃	[22]
13-2-34	12ξ-acetoxy-11β-hydroxypetuniasterone M-7-acetate	C ₃₅ H ₅₀ O ₉	CDCl ₃	[22]
13-2-35	petuniasterone M	C ₃₁ H ₄₆ O ₅	CDCl ₃	[22]
13-2-36	12α-acetoxypetuniasterone M	C ₃₃ H ₄₈ O ₇	CDCl ₃	[22]
13-2-37	24- <i>epi</i> -cyclocitrinol	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-38	cyclocitrinol	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-39	20- <i>O</i> -methyl-24- <i>epi</i> -cyclocitrinol	C ₂₆ H ₃₈ O ₄	CDCl ₃	[23]

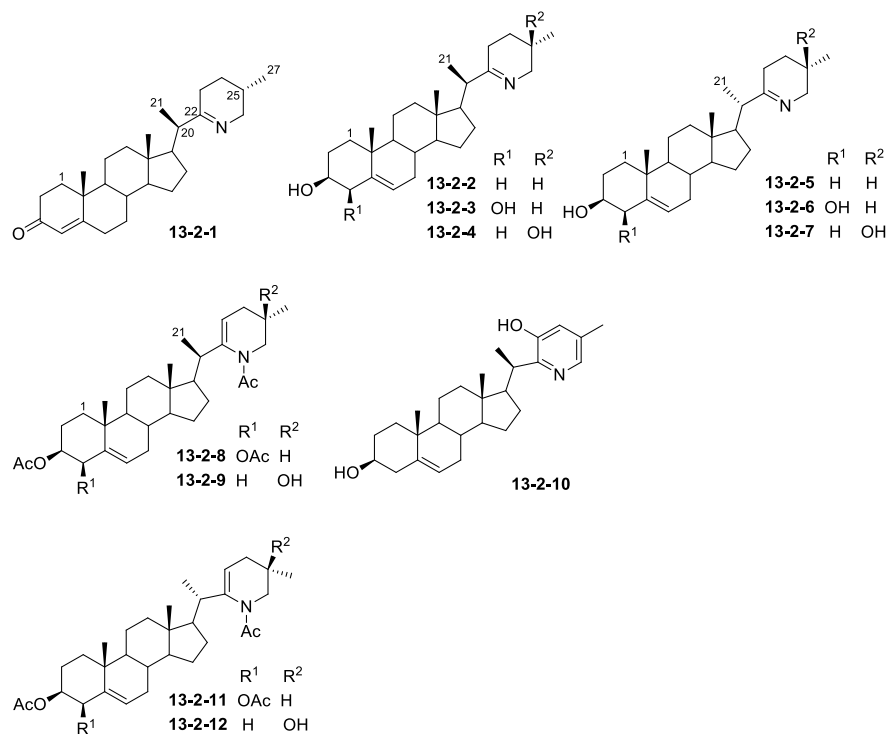
Table 13-2-1 (continued)

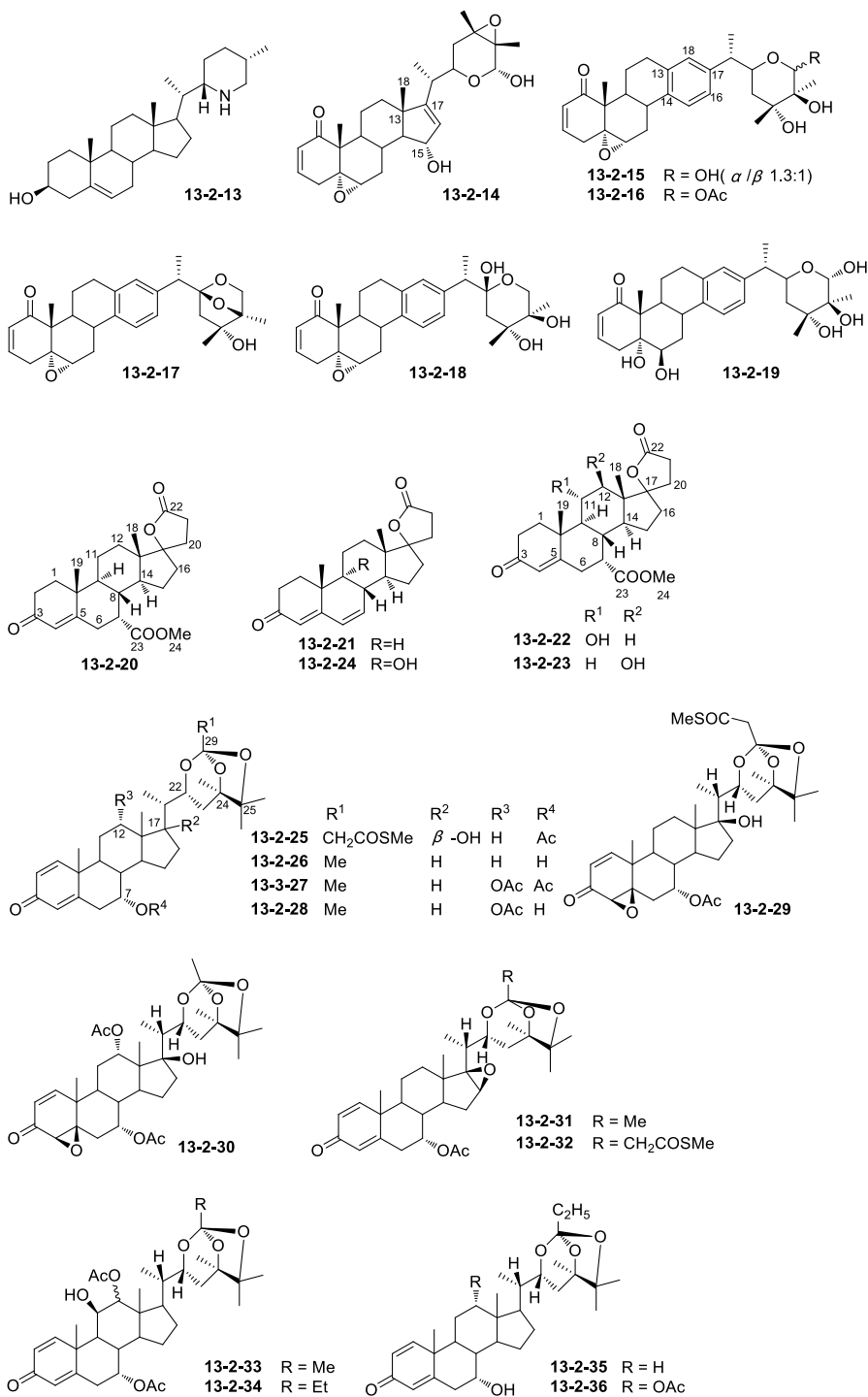
No.	Compounds	MFs	Test solvents	References
13-2-40	20- <i>O</i> -methylcyclocitrinol	C ₂₆ H ₃₈ O ₄	CDCl ₃	[23]
13-2-41	24-oxocyclocitrinol	C ₂₅ H ₃₄ O ₄	CDCl ₃	[23]
13-2-42	12 <i>R</i> -hydroxycyclocitrinol	C ₂₅ H ₃₆ O ₅	DMSO- <i>d</i> ₆	[23]
13-2-43	neocyclocitrinol A	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-44	neocyclocitrinol B	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-45	neocyclocitrinol C	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-46	neocyclocitrinol D	C ₂₅ H ₃₆ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-47	<i>erythro</i> -23- <i>O</i> -methylneocyclocitrinol	C ₂₆ H ₃₈ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-48	<i>threo</i> -23- <i>O</i> -methylneocyclocitrinol	C ₂₆ H ₃₈ O ₄	DMSO- <i>d</i> ₆	[23]
13-2-49	isocyclocitrinol B	C ₂₅ H ₃₆ O ₄	CDCl ₃	[23]
13-2-50	precyclocitrinol B	C ₂₅ H ₃₄ O ₃	CDCl ₃	[23]

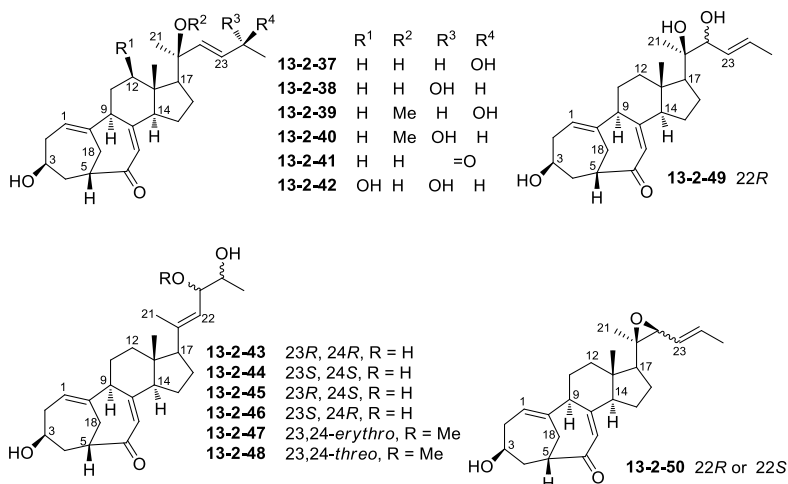
① Acetylated product.

② Reduced product.

③ Synthetic compound.





**Table 13-2-2:** ¹H NMR spectroscopic data of steroids **13-2-1**~**13-2-4**.

H	13-2-1	13-2-2	13-2-3	13-2-4
3		3.48 m	3.52 dt(3.3, 12.2)	3.34 m
4	5.71 br s		4.09 d(2.8)	
6		5.33 br d(5.3)	5.62 br d(3.7)	5.20 d(4.9)
18	0.74 s	0.70 s	0.69 s	0.62 s
19	1.16 s	0.98 s	1.15 s	1.05 s
21	0.97 d(6.9)	0.97 d(7.8)	0.96 d(6.9)	0.91 d(6.9)
26ax	2.92 m	2.93 m	2.92 m	3.23 m
26eq	3.69 m	3.69 m	3.68 m	3.23 m
27	0.91 d(6.6)	0.90 d(6.6)	0.90 d(6.6)	0.91 s

Table 13-2-3: ¹H NMR spectroscopic data of steroids **13-2-5**~**13-2-8**.

H	13-2-5	13-2-6	13-2-7	13-2-8
3	3.05 m	3.55 dt(3.6, 11.6)	3.34 m	4.73 dt(3.3, 12.2)
4		4.11 d(3.2)		5.49 d(2.7)
6	5.32 br d(5.0)	5.65 br d(3.5)	5.20 d(4.9)	5.80 br d(3.6)
18	0.69 s	0.69 s	0.60 s	0.61 s
19	0.99 s	1.17 s	0.88 s	1.11 s
21	1.07 d(6.7)	1.07 d(6.6)	1.01 d(6.8)	1.14 d(6.9)
23				5.11 t(3.4)
26ax	2.96 m	2.97 m	3.23 m	
26eq	3.65 dd(6.7, 16.6)	3.66 m	3.23 m	
27	0.88 d(6.7)	0.90 d(6.9)	1.08 s	0.96 d(6.6)
Ac				2.00 s, 2.06 s, 2.15 s

Table 13-2-4: ^1H NMR spectroscopic data of steroids **13-2-9~13-2-13**.

H	13-2-9	13-2-10	13-2-11	13-2-12	13-2-13
3	4.61 m	3.38 m	4.74 dt(3.6, 11.6)	4.62 m	3.52 m
4			5.49 d(2.8)		
6	5.48 br d(5.0)	5.22 br d(4.9)	5.80 m	5.37 br d(4.8)	5.33 br d(4.7)
18	0.63 s	0.73 s	0.67 s	0.69 s	0.68 s
19	1.28 s	0.92 s	1.13 s	1.25 s	1.00 s
21	1.18 d	1.14 d(6.6)	1.12 d(5.7)	1.11 d	0.85 d(6.4)
22					2.65 br d(11.4)
23	5.09 t(3.1)		5.18 m	5.16 t(3.2)	
24		6.79 br s			
26ax		7.71 br s			2.26 m
26eq					3.22 br d(11.4)
27	1.00 s	2.12 s	0.94 d(6.6)	1.02 s	1.03 d(6.7)
Ac	2.01 s, 2.22 s		2.01 s, 2.06 s, 2.14 s	2.20 s, 2.02 s	

Table 13-2-5: ^1H NMR spectroscopic data of steroids **13-2-14~13-2-18**.

H	13-2-14	13-2-15	13-2-16	13-2-17	13-2-18
2	5.99 dd(10.1, 2.3)	6.00 dd(10.0, 2.6)	6.00 dd(10.0, 2.5)	6.00 dd(10.2, 2.2)	6.00 dd(10.1 2.2)
3	6.70 ddd (10.1, 5.0, 2.3)	6.76 ddd (10.0, 5.0, 2.6)	6.75 ddd (10.0, 5.0, 2.5)	6.76 ddd (10.2, 5.0, 2.2)	6.75 ddd (10.1, 5.0, 2.2)
4 α	1.85 dd(19.5, 5.0)	1.90 dd(19.2, 5.0)	1.90 dd(19.2, 5.0)	1.90 dd(19.6, 5.0)	1.90 dd(19.6, 5.0)
4 β	3.12 dt(19.5, 2.3)	3.13 dt(19.2, 2.6)	3.13 dt(19.2, 2.5)	3.14 dt(19.6, 2.2)	3.15 dt(19.6, 2.2)
6	3.08 d(5.0)	3.22 d(4.6)	3.23 d(4.8)	3.22 d(4.8)	3.23 d(4.0)
7 α	1.70 m	1.90 m		1.84 m	1.84 m
7 β	2.15 m	2.70 m		2.65 m	2.65 m
14	1.20 m				
15	4.51 d(8.5)	7.11 d(8.0)	7.10 d(8.0)	7.10 br s	7.12 br s
16	5.39 br s	6.98 dd(8.0, 1.0)	6.99 dd(8.0, 1.0)	7.10 br s	7.12 br s
18	0.82 s	6.91 d(1.0)	6.94 d(1.0)	6.99 br s	7.00 br s
19	1.40 s	1.38 s	1.38 s	1.37 s	1.38 s
20	2.10 m	2.78 m		3.05 q(7.0)	2.81 q(7.0)
21	1.01 d(7.1)	1.23 d(7.0)	1.25 d(6.9)	1.36 d(7.0)	1.35 d(7.0)
22	3.70 ddd(10.6, 6.9, 3.7)	4.13 m	3.80 m		
23 α	1.85 m	1.55 m		1.73 d(12.9)	1.54 bs
23 β	1.55 m	1.73 m		1.70 d(12.9)	1.54 bs
26 α	4.96 d(9.0)	4.92 br s	5.57 s	4.38 d(6.7)	4.12 d(12.1)
26 β				3.31 d(6.7)	3.28 d(12.1)
27	1.41 s	1.43 s	1.24 s	1.35 s	1.19 s
28	1.39 s	1.38 s	1.38 s	1.25 s	1.12 s
16-OH	3.45 d(9.0)				
OAce			2.14 s		

Table 13-2-6: ^1H NMR spectroscopic data of steroids **13-2-19~13-2-21**.

H	13-2-19	13-2-20	13-2-21
1		1.82 m, 2.23 m	2.01 dd(11.8, 4.7) 1.68 m
2	5.86 dd(10.0, 2.4)	2.41 m	2.42 m, 2.55 m
3	6.69 ddd(10.0, 5.2, 2.4)		
4	α 2.13 dd(20.0, 5.2) β 3.25 dt(20.0, 2.4)	5.69 s	5.65 br s
6	3.78 br s	2.62 ddd(14.9, 6.0, 1.9) 2.47 m	6.11 dd(9.9, 2.3)
7		2.82 td(4.1, 1.9)	6.05 d(10.2)
8		1.86 m	2.28 m
9		1.78 m	1.18 ddd(10.0, 4.2, 2.8)
11		1.40 m, 1.66 m	1.46 dd(12.5, 3.1), 1.66 m
12		1.33 dd(12.2, 3.7), 1.56 dt(11.7, 3.1)	1.36 m, 1.63 m
14		1.43 m	1.35 m
15	7.20 d(8.0)	1.75 m, 1.41 m	1.87 m, 1.59 m
16	7.00 dd(8.0, 1.0)	2.05 ddd(13.2, 4.9, 4.7) 1.84 m	2.29 m 1.86 m
18	6.98 d(1.0)	0.95 s	1.01 s
19	1.38 s	1.20 s	1.11 s
20		2.38 m, 1.89 m	2.34 m, 1.90 m
21	1.23 d(7.0)	2.52 m, 2.52 m	2.53 m, 2.47 m
22	4.13 m		
26	4.89 br s		
27	1.45 s		
28	1.38 s		
COOMe		3.61 s	

Table 13-2-7: ^1H NMR spectroscopic data of steroids **13-2-22~13-2-24**.

H	13-2-22	13-2-23	13-2-24
1	2.76 dd(14.5, 3.5) 1.98 dd(14.0, 4.5)	2.02 m, 2.47 m	1.77 m, 2.36 dd(13.8, 5.3)
2	2.43 m, 2.30 m	2.38 m, 2.34 m	2.54 m, 2.48 m
4	5.65 s	5.69 s	5.84 br s
6	2.60 ddd(14.6, 5.8, 1.7)	2.61 ddd(14.9, 6.1, 1.7)	6.27 dd(9.8, 2.9)
6	2.48 m	2.41 m	
7	2.78 m	2.81 m	5.91 dd(9.9, 1.9)
8	1.75 ddd(11.3, 11.2, 4.1)	1.78 m	2.63 dm(12.3)
9	1.89 m	1.87 m	
11	4.01 ddd(10.3, 10.2, 4.6)	1.78 m, 1.41 m	1.72 m, 1.65 m
12	1.38 m, 1.82 m	3.68 dd(10.7, 4.6)	1.38 ddd(11.8, 3.4, 2.9) 1.38 ddd(11.8, 3.4, 2.9)

Table 13-2-7 (continued)

H	13-2-22	13-2-23	13-2-24
14	1.62 m	1.39 m	1.97 m
15	1.70 m, 1.37 m	1.76 m, 1.48 m	1.83 m, 1.55 dd(12.2, 5.7)
16	1.83 m, 2.22 ddd(14.6, 12.2, 3.5)	1.79 m, 2.26 dd(13.7, 3.2)	1.88 m, 2.32 m
18	0.97 s	0.97 s	1.02 s
19	1.33 s	1.20 s	1.21 s
20	2.36 m, 1.92 m	2.47 m, 1.92 m	2.47 m, 1.95 m
21	2.53 m, 2.46 m	2.72 m, 2.50 m	2.53 m, 2.49 m
COOMe	3.61 s	3.60 s	

Table 13-2-8: ¹H NMR spectroscopic data of steroids 13-2-25 and 13-2-26.

H	13-2-25	13-2-26	H	13-2-25	13-2-26
1	7.10 d(10)	7.08 d(10)	22	4.30 ddd(11, 7, 4)	4.20 dt(11, 5)
2	6.26 dd(10, 2)	6.25 dd(10, 2)	26	1.33 s	1.30 s
4	6.02 br s	6.14 br t(2)	27	1.14 s	1.16 s
6	2.60 d(14)	2.48 dd(14, 3)	28	1.20 s	1.20 s
	2.66 d(14)	2.74 ddd(14, 3, 2)			
7	5.08 br q(3)	4.044 br s	COSMe	2.31 s	
12β		2.03 dt(12.5, 4)	CH ₂ CO	3.02, 3.07 ABq(14)	
18	0.97 s	0.76 s	OAc	1.98 s	
19	1.26 s	1.24 s	Orthoacetate-H ₃		1.56 s
21	0.91 d(7)	0.96 d(7)			

Table 13-2-9: ¹H NMR spectroscopic data of steroids 13-2-27~13-2-31.

H	13-2-27	13-2-28	13-2-29	13-2-30	13-2-31
1	6.96 d(10)	6.95 d(10)	6.55 d(11)	6.44 d(11)	7.05 d(10)
2	6.25 dd(10, 2)	6.23 dd(10, 2)	5.90 dd(11, 2)	5.86 dd(11, 2)	6.28 dd(10, 2)
4	6.03 br d(2)	6.14 br t(2)	3.12 d(2)	3.10 d(2)	6.00 br d(2)
6	2.60 d(14)	2.50 dd(14, 3)	2.36 dd(14, 2.5)	2.34 dd(15, 3)	2.60 d(3)
	2.66 d(14)	2.76 ddd(14, 3, 2)	1.54 dd(14, 2.5)	ca. 1.55	
7	5.08 br s	4.06 br s	5.12 br q(2.5)	5.12 br t(3)	4.95 br s
12	β 5.08 br s	β 5.07 br t(3)		5.04 br s	
16					3.34 d(4)
18	0.83 s	0.83 s	0.94 s	0.81 s	0.93 s
19	1.23 s	1.22 s	1.33 s	1.30 s	1.24 s

Table 13-2-9 (continued)

H	13-2-27	13-2-28	13-2-29	13-2-30	13-2-31
20			1.84 m		2.20 br m
21	0.85 d(7)	0.86 d(7)	0.90 d(7)	0.84 d(7)	0.81 d(7)
22	4.16 td(3, 4)	4.18 td(8, 4)	4.29 ddd (11, 8, 4)	4.16 td(8, 3.5)	4.26 ddd (11.5, 6, 4.5)
23	1.50 d(8)	1.50 d(8)	1.62 m(4, 11)	1.50 d(8)	
26	1.30 s	1.30 s	1.33 s	1.30 s	1.34 s
27	1.16 s	1.16 s	1.14 s	1.16 s	1.18 s
28	1.18 s	1.18 s	1.20 s	1.19 s	1.19 s
Orthoacetate-H ₃	1.54 s	1.56 s		1.54 s	1.55 s
OAc	2.03 s, 2.08 s	2.04 s	2.03 s	2.07 s, 2.08 s	2.01 s
COSMe			2.31 s		
CH ₂ CO			3.01, 3.06 ABq(14)		

Table 13-2-10: ¹H NMR spectroscopic data of steroids 13-2-32~13-2-36.

H	13-2-32	13-2-33	13-2-34	13-2-35	13-2-36
1	7.05 d(10)	7.20 d(10)	7.20 d(10)	7.08 d(10)	6.95 d(10)
2	6.28 dd(10, 2)	6.30 dd(10, 2)	6.30 dd(10, 2)	6.26 dd(10, 2)	6.22 dd(10, 2)
4	6.00 br d(2)	5.98 br s	5.98 br s	6.14 brt(2)	6.14 brt(2)
6	2.60 d(3)	α 2.56 dd(14, 3) β 2.74 ddd(14, 3, 2)	α 2.56 dd(14, 3) β 2.74 ddd(14, 3, 2)	α 2.48 dd(14, 3) β 2.75 ddd(14, 3, 2)	α 2.50 dd(14, 3) β 2.75 ddd(14, 3, 2)
7	4.94 br s	5.18 br q(3)	5.18 br q(3)	4.05 br s	4.06 br s
8		2.20 td(12, 3)	2.20 td(12, 3)		
9		1.65 dd(12, 3)			
11		4.22 m	4.22 m		
12		4.86 d(3)	4.87 d(3)	β 2.04 dt(12.5, 4)	β 5.06 brt(3)
16	3.33 d(4)				
18	0.93 s	1.04 s	1.04 s	0.76 s	0.83 s
19	1.24 s	1.48 s	1.48 s	1.23 s	1.21 s
20	2.20 br m	1.72			
21	0.82 d(7)	0.84 d(6)	0.83 d(6.5)	0.97 d(7)	0.84 d(7)
22	4.31 ddd(11, 6, 5)	4.16 td(8, 4)	4.16 td(8, 4)	4.20 dt(11.5)	4.18 td(8, 4)
23					1.47 d(8)
26	1.36 s	1.30 s	1.32 s	1.31 s	1.31 s
27	1.13 s	1.17 s	1.16 s	1.16 s	1.14 s
28	1.21 s	1.19 s	1.18 s	1.19 s	1.18 s
COSMe	2.30 s				
CH ₂ CO	3.02, 3.09 ABq(14)				

Table 13-2-10 (continued)

H	13-2-32	13-2-33	13-2-34	13-2-35	13-2-36
Ortho- acetate-H ₃		1.55 s			
OAc	2.01 s	2.03 s, 2.09 s	2.04 s, 2.09 s		2.03 s
Orthopro- pionate-H ₂			1.80 m	1.80 m	1.80 m
Orthopro- pionate-H ₃			0.98 t(8)	0.99 t(7.5)	0.97 t(7.5)

Table 13-2-11: ¹H NMR spectroscopic data of steroids **13-2-37~13-2-41**.

H	13-2-37	13-2-38	13-2-39	13-2-40	13-2-41
1	5.53 dd(6.6, 8.1)	5.53 dd(6.8, 8.7)	5.56 dd(6.2, 8.4)	5.56 dd(6.2, 8.4)	5.57 dd(5.5, 8.2)
2 α	2.07 ddt (2.2, 8.1, 13.2)	2.07 ddt (2.1, 8.2, 13.3)	2.24 ddt (2.2, 8.4, 13.2)	2.24 ddt (2.6, 8.4, 13.2)	2.25 ddt (2.2, 8.7, 13.2)
2 β	2.33 ddd (6.6, 11.0, 13.2)	2.33 ddd (6.4, 11.0, 13.3)	2.48 ddd (6.2, 11.3, 13.2)	2.48 ddd (6.2, 11.3, 13.2)	2.48 ddd (5.9, 11.9, 13.2)
3	3.10 m	3.11 m	3.49 m	3.50 m	3.50 m
4 α	2.61 br d(13.2)	2.62 br d(13.2)	2.89 br d(12.8)	2.89 br d(12.8)	2.89 br d(12.8)
4 β	1.51 dd(3.7, 13.2)	1.51 dd(4.0, 13.2)	1.67 m	1.67 m	1.68 m
5	2.66 m	2.67 m	2.74 m	2.74 m	2.74 m
7	5.37 s	5.38 s	5.56 s	5.56 s	5.57 s
9	2.78 dd(5.8, 12.5)	2.78 dd(5.5, 12.4)	2.76 dd(5.8, 12.4)	2.76 dd(5.8, 12.4)	2.77 dd(5.9, 12.4)
11 α	1.49 m	1.49 m	1.58 m	1.58 m	1.61 m
11 β	1.74 m	1.75 m	1.67 m	1.67 m	1.80 m
12 α	1.42 td(4.4, 13.2)	1.42 m	1.43 m	1.43 m	1.51 m
12 β	2.13 m	2.13 m	2.19 m	2.19 m	2.20 m
14	2.10 ddd(1.7, 6.6, 12.1)	2.10 m	2.07 q(12.1)	2.07 q(12.1)	2.11 ddd(12.3, 6.9)
15 α	1.38 m	1.38 m	1.47 m	1.47 m	1.63 m
15 β	1.46 m	1.46 m	1.58 m	1.58 m	1.55 m
16 α	1.56 m	1.56 m	1.68 m	1.68 m	1.82 m
16 β	1.66 m	1.66 m	1.73 m	1.73 m	1.73 m
17	1.66 m	1.66 m	1.84 m	1.84 m	1.87 t(10.0)
18 α	2.46 br s	2.47 br s	2.56 m	2.56 m	2.53 m
18 β	2.50 m	2.50 m	2.57 m	2.57 m	2.55 m
19	0.69 s	0.71 s	0.74 s	0.74 s	0.78 s

Table 13-2-11 (continued)

H	13-2-37	13-2-38	13-2-39	13-2-40	13-2-41
21	1.20 s	1.20 s	1.29 s	1.29 s	1.41 s
22	5.62 d(15.3)	5.62 dd(1.3, 15.6)	5.67 d(15.7)	5.68 d(15.9)	6.89 d(15.5)
23	5.49 dd(5.9, 15.3)	5.49 dd(5.5, 15.6)	5.57 dd(6.2, 15.7)	5.57 dd(6.2, 15.9)	6.28 d(15.5)
24	4.10 m	4.10 m	4.35 m	4.35 m	
25	1.08 d(5.9)	1.08 d(6.4)	1.29 d(6.2)	1.29 d(6.2)	2.27 s
OMe			3.14 s	3.14 s	
3-OH	4.60 d(4.4)	4.61 d(4.5)			
24-OH	4.57 d(4.4)	4.57 d(4.5)			

Table 13-2-12: ^1H NMR spectroscopic data of steroids **13-2-42~13-2-44**.

H	13-2-42	13-2-43	13-2-44
1	5.54 m	5.55 dd(6.4, 8.2)	5.55 dd(6.4, 8.2)
2 α	2.06 ddt(2.3, 8.7, 13.2)	2.07 m	2.07 m
2 β	2.33 ddd(6.4, 11.0, 13.2)	2.34 m	2.34 m
3	3.11 m	3.12 m	3.12 m
4 α	2.61 br d(12.8)	2.63 br d(12.8)	2.63 br d(13.3)
4 β	1.51 m	1.52 m	1.52 m
5	2.67 m	2.68 m	2.68 m
7	5.41 s	5.42 s	5.42 s
9	2.93 brt(8.7, 9.7)	2.84 dd(6.0, 12.4)	2.84 dd(5.5, 11.9)
11 α	1.58 m	1.54 m	1.54 m
11 β	1.59 m	1.76 m	1.80 m
12 α	3.52 brt(7.3, 8.7)	1.43 td(4.4, 12.8)	1.47 td(4.6, 12.8)
12 β		1.74 m	1.74 m
14	2.12 m	2.22 brt(8.7)	2.25 brt(8.7)
15 α	1.46 m	1.57 m(8.7)	1.57 m
15 β	1.56 m	1.52 m(8.7)	1.52 m
16 α	1.54 m	1.82 m	1.84 m
16 β	1.74 m	1.68 m	1.65 m
17	1.84 t(9.6)	2.27 brt(10.1)	2.23 brt(9.6)
18 α	2.41 dd(6.4, 7.8)	2.47 br s	2.47 br s
18 β	2.47 br s	2.47 br s	2.47 br s
19	0.55 s	0.53 s	0.50 s
21	1.12 s	1.66 s	1.66 s
22	5.77 dd(1.3, 15.6)	5.15 d(8.7)	5.16 d(8.7)
23	5.53 dd(5.5, 15.6)	3.95 ddd(4.0, 6.6, 8.7)	3.95 ddd(4.0, 6.6, 8.7)
24	4.11 m	3.40 m	3.40 m
25	1.09 d(6.4)	0.96 d(6.4)	0.94 d(6.4)
3-OH	4.61 d(4.1)	4.62 d(4.6)	4.62 d(4.6)
12-OH	6.03 d(1.8)		
23-OH		4.47 d(4.0)	4.49 d(4.0)
24-OH	4.58 d(4.6)	4.38 d(4.0)	4.37 d(4.0)

Table 13-2-13: ^1H NMR spectroscopic data of steroids **13-2-45~13-2-47**.

H	13-2-45	13-2-46	13-2-47
1	5.55 dd(6.4, 8.2)	5.55 dd(6.6, 8.2)	5.55 dd(6.4, 8.2)
2 α	2.07 m	2.07 m	2.07 m
2 β	2.34 m	2.34 m	2.34 m
3	3.12 m	3.12 m	3.12 m
4 α	2.63 br d(12.8)	2.63 br d(12.8)	2.63 br d(12.7)
4 β	1.54 m	1.52 m	1.52 m
5	2.68 m	2.68 m	2.67 m
7	5.42 s	5.42 s	5.42 s
9	2.84 dd(5.9, 11.5)	2.84 dd(5.5, 12.0)	2.84 dd(5.5, 11.9)
11 α	1.54 m	1.54 m	1.54 m
11 β	1.79 m	1.70 m	1.80 m
12 α	1.43 td(4.6, 12.4)	1.47 td(4.2, 12.4)	1.47 m
12 β	1.74 m	1.74 m	1.74 m
14	2.25 brt(8.8)	2.25 brt(8.7)	2.25 brt(8.7)
15 α	1.57 m	1.54 m	1.54 m
15 β	1.52 m	1.49 m	1.49 m
16 α	1.82 m	1.82 m	1.82 m
16 β	1.68 m	1.68 m	1.68 m
17	2.26 brt(9.6)	2.23 brt(9.6)	2.31 brt(9.6)
18 α	2.48 br s	2.47 br s	2.47 br s
18 β	2.48 br s	2.47 br s	2.47 br s
19	0.53 s	0.50 s	0.52/0.53 s
21	1.64 s	1.65 s	1.68 s
22	5.22 d(8.7)	5.22 d(8.2)	5.11 d(9.1)
23	4.05 ddd(4.6, 4.6, 8.7)	4.09 ddd(4.6, 5.0, 8.2)	3.78/3.77 dd(4.1, 9.1)
24	3.50 m	3.49 m	3.60 m
25	0.97 d(6.4)	0.96 d(6.4)	0.96/0.98 d(6.4)
OMe			3.15/3.16 s
3-OH	4.62 d(4.6)	4.62 d(4.6)	4.62 d(4.0)
23-OH	4.38 d(4.6)	4.41 d(5.0)	
24-OH	4.29 d(5.0)	4.29 d(4.6)	4.42 d(5.0)

Table 13-2-14: ^1H NMR spectroscopic data of steroids **13-2-48~13-2-50**.

H	13-2-48	13-2-49	13-2-50
1	5.55 dd(6.4, 8.2)	5.57 brt(7.3)	5.57 dd
2 α	2.07 m	2.25 ddt(13.3, 8.2)	2.25 ddt(13.3, 8.2, 2.2)
2 β	2.34 m	2.48 ddd(13.3, 11.4, 6.4)	2.49 ddd(13.3, 11.4, 6.0)
3	3.12 m	3.50 brt(11.0)	3.51 brt(11.4)
4 α	2.62 br d(13.4)	2.89 br d(12.8)	2.89 br d(13.1)
4 β	1.52 m	1.68 m	1.68 m
5	2.67 m	2.75 m	2.75 m

Table 13-2-14 (continued)

H	13-2-48	13-2-49	13-2-50
7	5.42 s	5.59 s	5.59 s
9	2.84 dd(5.9, 11.9)	2.78 dd(12.8, 6.0)	2.78 dd(12.4, 5.9)
11 α	1.54 m	1.61 m	1.61 m
11 β	1.80 m	1.85 m	1.89 m
12 α	1.47 m	1.48 m	1.51 m
12 β	1.74 m	2.17 m	2.15 m
14	2.25 brt(8.7)	2.09 ddd(12.3, 6.4)	2.14 m
15 α	1.54 m	1.62 m	1.63 m
15 β	1.49 m	1.54 m	1.54 m
16 α	1.82 m	1.94 m	1.68 m
16 β	1.68 m	1.74 m	1.62 m
17	2.31 brt(9.6)	1.74 m	1.85 t(9.6)
18 α	2.47 br s	2.55 d(13.3)	2.57 d(13.2)
18 β	2.47 br s	2.59 dd(13.3, 6.0)	2.59 dd(13.2, 6.4)
19	0.52/0.54 s	0.86 s	0.78 s
21	1.71 s	1.26 s	1.33 s
22	5.01 d(9.6)	3.91 d(7.8)	3.09 d(7.4)
23	3.71 dd(6.4, 9.6)	5.41 ddq(15.1, 8.2, 1.4)	5.37 ddq(15.4, 7.4, 1.3)
24	3.53 m	5.78 dqd(15.1, 6.4)	5.88 dqd(15.4, 6.8)
25	0.93/0.95 d(6.6)	1.74 dd(6.4, 0.9)	1.76 dd(6.8, 1.3)
OMe	3.14/3.15 s		
3-OH	4.62 d(4.5)		
24-OH	4.44/4.43 d(4.1)		

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14 Tropolones and cerebrosides

14.1 Tropolones

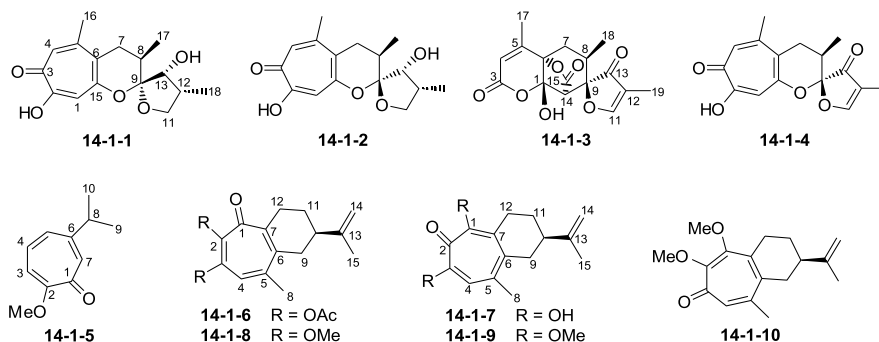
Table 14-1-1: Compounds, MFs, and test solvents of tropolones **14-1-1~14-1-23**.

No.	Compounds	MFs	Test solvents	References
14-1-1	malettinin B	C ₁₆ H ₂₀ O ₅	CD ₃ COCD ₃	[1]
14-1-2	malettinin C	C ₁₆ H ₂₀ O ₅	CD ₃ COCD ₃	[1]
14-1-3	malettinin D	C ₁₆ H ₁₆ O ₇	CD ₃ OD	[1]
14-1-4	malettinin A	C ₁₆ H ₁₆ O ₅	CDCl ₃	[2]
14-1-5 ^①	2-methoxy-6-(methylethyl)cyclohepta-2,4,6-trien-1-one	C ₁₁ H ₁₄ O ₂	CDCl ₃	[3]
14-1-6 ^①	–	C ₁₉ H ₂₂ O ₅	CDCl ₃	[4]
14-1-7	–	C ₁₅ H ₁₈ O ₃	CDCl ₃	[4]
14-1-8 ^①	–	C ₁₇ H ₂₂ O ₃	CDCl ₃	[4]
14-1-9 ^①	–	C ₁₇ H ₂₂ O ₃	CDCl ₃	[4]
14-1-10 ^①	–	C ₁₇ H ₂₂ O ₃	CDCl ₃	[4]
14-1-11	multipolone	C ₁₉ H ₂₄ O ₃	–	[5]
14-1-12 ^②	pycnidione	C ₃₃ H ₄₀ O ₇	CDCl ₃	[6]
14-1-13 ^②	epolone A	C ₃₂ H ₄₀ O ₆	CD ₃ OD	[6]
14-1-14	epolone B	C ₂₄ H ₃₂ O ₄	CDCl ₃	[6]
14-1-15	colchicone	C ₂₀ H ₂₀ O ₆	–	[7]
14-1-16	3-demethylcolchicone	C ₁₉ H ₁₈ O ₆	–	[7]
14-1-17	cornigerone	C ₁₉ H ₁₆ O ₆	–	[7]
14-1-18	noreupenifeldin	C ₃₂ H ₄₀ O ₆	CDCl ₃	[8]
14-1-19	3,6,9-trimethoxyphenanthropolone ^③	C ₂₀ H ₂₀ O ₄	CDCl ₃	[9]
14-1-20	4,6,9-trimethoxyphenanthropolone ^③	C ₂₀ H ₂₀ O ₄	CDCl ₃	[9]
14-1-21	colchicine	C ₂₂ H ₂₅ NO ₆	CDCl ₃	[10]
14-1-22	2,3-didemethylcolchicine	C ₂₀ H ₂₁ NO ₆	CD ₃ OD	[11]
14-1-23	1,2-didemethylcolchicine	C ₂₀ H ₂₁ NO ₆	CD ₃ OD	[11]

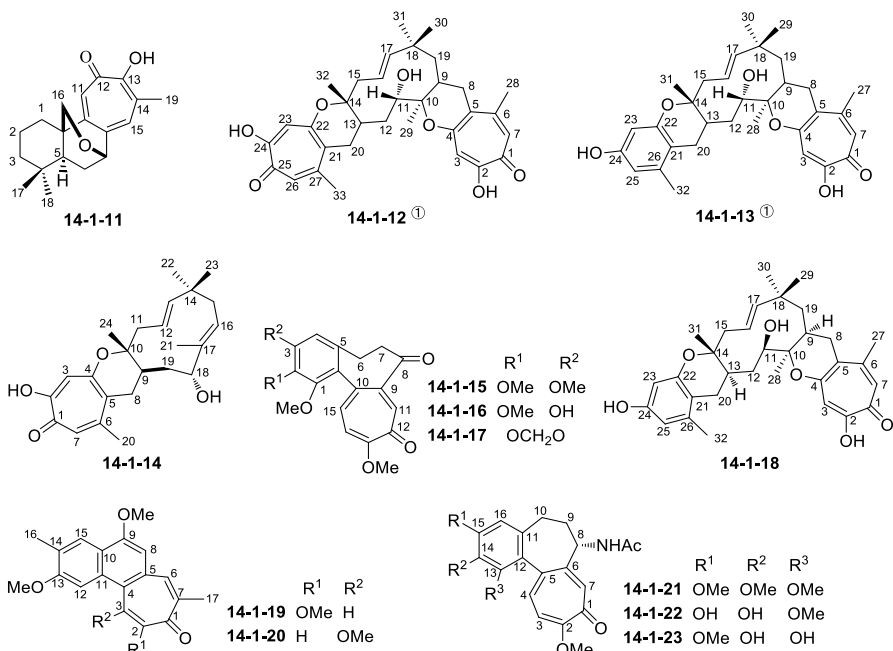
^① Synthetic compounds.

^② The displayed structures of **14-2-12** and **14-2-13** in the original literature seem to be mistaken.

^③ In this handbook, the atomic numbers of **14-2-19** and **14-2-20** are different from those of [9], but the names in [9] are used.



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^① The displayed structures of **14-2-12** and **14-2-13** in the original literature seem to be mistaken.

Table 14-1-2: ¹H NMR spectroscopic data of tropolones **14-1-1**~**14-1-5**.

H	14-1-1	14-1-2	14-1-3	14-1-4	14-1-5
1	6.76 s	6.95 s		6.96 s	
2					3.92 s(OMe)
3					6.65 d(10.0)
4	7.01 s	7.06 s	6.22 q(1.8)	7.20 s	7.01 dd(10.0, 11.1)
5					6.79 dd(1.7, 11.1)
7	3.02 dd(17, 6.2) 2.56 (17, 3.8)	2.80 dd(17, 5.9) 2.52 dd(17, 13)	2.55 dd(14, 6.3) 2.05 dd(14, 11)	eq 2.75 dd(17, 5.7) ax 2.52 dd(17, 13)	7.18 d(1.7)
8	2.57 m	2.22 m	2.42 m	2.36 m	2.78 m(6.8)
9					1.23 d(6.8)
10					1.23 d(6.8)
11	4.18 t(7.8) 3.64 dd(10, 7.8)	4.12 dd(8.8, 7.7) 3.65 dd(8.8, 5.8)	8.32 q(1.2)	8.02 q(1.1)	
12	2.70 m	2.50 m			
13	3.90 d(4.8)	4.32 d(8.7)			
14			2.76 s		
16	2.36 s	2.40 s		2.39 s	
17	1.01 d(6.6)	1.08 d(6.6)	2.10 d(1.8)	0.95 d(6.5)	
18	1.04 d(6.6)	1.21 d(7.2)	0.81 d(6.7)	1.77 d(1.1)	
19			1.72 d(1.2)		

Table 14-1-3: ^1H NMR spectroscopic data of tropolones **14-1-6**~**14-1-10**.

H	14-1-6	14-1-7	14-1-8	14-1-9	14-1-10
4	6.84	7.42	6.75	6.73	7.02
=CH ₂	4.74, 4.79	4.76, 4.81	4.75, 4.80	4.74, 4.78	4.74, 4.79
OMe			3.81, 3.90	3.82, 3.90	3.82, 3.85
Ac	2.33, 2.33				
8-Me	2.30	2.41	2.30	2.38	2.26
15-Me	1.78	1.82	1.80	1.80	1.79

Table 14-1-4: ^1H NMR spectroscopic data of tropolones **14-1-11**~**14-1-14** and **14-1-18**.

H	14-1-11	14-1-12	14-1-13	14-1-14	14-1-18
1	α 1.95 dt(4.5, 13.4) β 1.72 dt d(13.4, 3, 1.9)				
2	1.62 m, 1.70 m				
3	α 1.20 dt(4, 13) β 1.60 dt d(1.9, 3.6, 13)	6.97 s	7.07 s	6.97 s	7.09 s
5	1.23 ddd(2.0, 6.2, 12.0)				
6	α 1.66 ddd(2.0, 12.0, 13.9) β 2.11 ddd(3.8, 6.2, 13.9)				
7	4.68 dd(2.0, 3.8)	7.10 s	7.09 s	7.10 s	7.29 s
8		2.63 m, 2.31 m	2.74 dd(18, 5.5) 2.48 dd(18, 5.5)	2.88 dd(17, 5.5) 2.36 dd(17, 12.5)	α 2.70 dd(16.0, 4.5) β 2.32 dd(16.0, <2.0)
9		1.65 m	1.78 m	1.64 m	1.75 m
11	7.40 s	3.60 br d(10)	3.66 br d(10)	2.56 br d(14.5) 2.23 br d(14.5, 10)	4.19 dd(11.0, <1)
12		1.60 br dd(15, 10) 1.14 m	1.70 br dd(15, 10) 1.12 m	5.04 ddd(15.5, 10, 3)	α 2.23 m β 1.55 ddd(13.0, 11.0, <2.0)
13	9.6 br(OH)	1.85 m	1.89 m	5.14 dd(15.5, 2)	2.19 m
15	7.28 s	2.69 dd, 2.30 dd	2.62 bd(14, 1.2) 2.38 dd(14, 11)	2.22 dd(12.5, 6) 1.77 dd(12.5, 5)	α 2.54 dd(13.5, 11.0) β 2.75 dd(13.5, 3.5)

Table 14-1-4 (continued)

H	14-1-11	14-1-12	14-1-13	14-1-14	14-1-18
16	α 2.99 dd(2.0, 9.1) β 4.36 d(9.1)	5.38 m	5.52 ddd(15, 9, 4)	5.20 dd(6, 5)	5.65 ddd(16.0, 11.0, 3.5)
17	1.16 s	5.36 m	5.47 d(15)		5.82 d(16.0)
18	0.86 s			3.94 bd(10)	
19	2.42 s	1.60 m	1.65 m	1.72 dd(14, 10)	α 0.83 dd(14.5, 4.5)
		1.14 m	1.13 m	1.12 bdd(14, 8)	β 1.78 dd(14.5, <2.0)
20		2.98 br d(17.5) 2.26 br d(17)	2.96 dd(16.5, 5.5) 2.24 br d(16.5)	2.39 s	α 2.44 m β 3.45 dd(17.58, 12.5)
21				1.63 s	
22				1.06 s	
23		6.93 s	6.06 s	0.99 s	6.17 s
24				1.10 s	
25			6.17 s		6.33 s
26		7.09 s			
27			2.43 s		2.46 s
28		2.36 s	1.24 s		1.09 s
29		1.24 s	1.12 s		1.05 s
30		1.05 s	1.05 s		1.10 s
31		1.08 s	1.17 s		1.42 s
32		1.20 s	2.15 s		2.16 s
33		2.39 s			

Table 14-1-5: ^1H NMR spectroscopic data of tropolones 14-1-15~14-1-17, 14-1-19, and 14-1-20.

H	14-1-15	14-1-16	14-1-17	14-1-19	14-1-20
1	3.55 s(OMe)	3.53 s(OMe)	3.79 s(OMe)		
2	3.86 s(OMe)	3.94 s(OMe)		4.06 s(OMe)	7.64 s
3	3.87 s(OMe)			7.98 s	3.93 s(OMe)
4	6.54 s	6.62 s	6.50 s		
6	α 3.11 ddd(5.4, 13.6, 13.7) β 2.67 ddd(2.2, 5.0, 13.7)	α 3.10 ddd(13.8, 5.3, 13.4) β 2.67 ddd(13.8, 4.9, 2.3)	α 3.05 ddd(5.2, 13.4, 13.6) β 2.65 ddd(2.3, 4.9, 13.6)	7.84 s	7.62 s
7	α 2.95 ddd(5.4, 2.2, 16.6) β 2.82 ddd(5.0, 13.6, 16.6)	α 2.95 ddd(5.3, 2.3, 16.6) β 2.82 ddd(13.4, 4.9, 16.6)	α 2.93 ddd(5.2, 2.3, 16.5) β 2.81 ddd(13.4, 4.9, 16.5)		
8				6.80 s	6.57 s

Table 14-1-5 (continued)

H	14-1-15	14-1-16	14-1-17	14-1-19	14-1-20
9				4.09 s(OMe)	4.01 s(OMe)
11	7.12 s	7.15 s	7.14 s		
12				7.63 s	7.32 s
13	4.00 s(OMe)	4.02 s(OMe)	4.02 s(OMe)	4.05 s(OMe)	3.94 s(OMe)
14	6.85 d(10.7)	6.85 d(10.6)	6.85 d(10.7)		
15	7.24 d(10.7)	7.24 d(10.6)	7.19 d(10.7)	8.13 d(0.7)	8.02 s
16			5.98 d(1.4)	2.48 d(0.5)	2.41 s
			5.99 d(1.4)		
17				2.45 s	2.41 s

Table 14-1-6: ¹H NMR spectroscopic data of tropolones 14-1-21~14-1-23.

H	14-1-21	14-1-22	14-1-23
2	4.03 s(OMe)	3.86 s(OMe)	
3	6.93 d(11.0)	7.045 d(10.5)	6.98 d(10.5)
4	7.39 d(11.0)	7.315 d(10.5)	7.23 d(10.5)
7	7.69 s	7.252	7.152
8	4.66 dt(11.8, 6, 5.8)	4.48 dd(6, 12)	4.39 dd(6,11.5)
9	2.38 m, 2.01 m	2.11, 1.89	1.94, 2.18
10	2.54 m	2.42, 2.21	2.28, 2.45
13	3.67 s(OMe)	3.515 s(OMe)	
14	3.95 s(OMe)		
15	3.92 s(OMe)		3.86 s(OMe)
16	6.55 s	6.46	6.43
NH	8.64 d(6.0)		
Ac	1.96 s	1.95	1.88

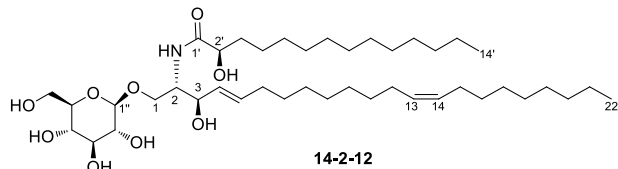
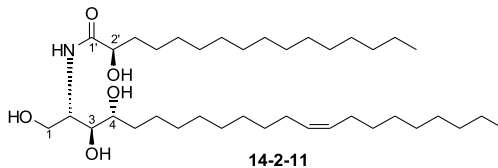
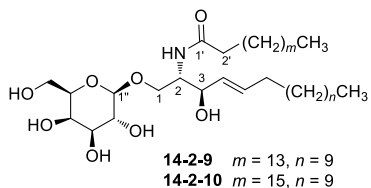
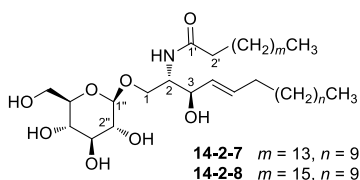
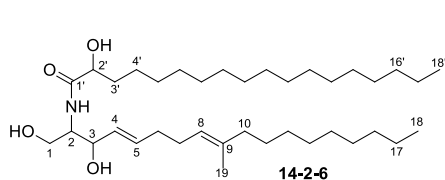
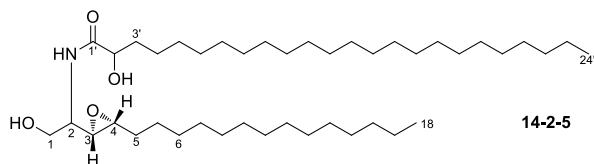
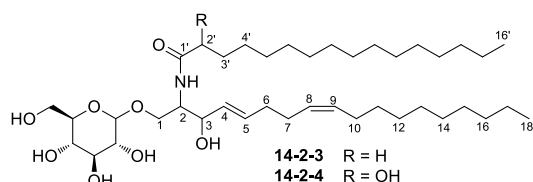
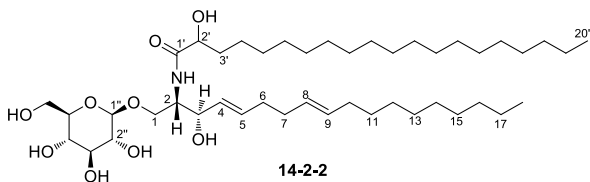
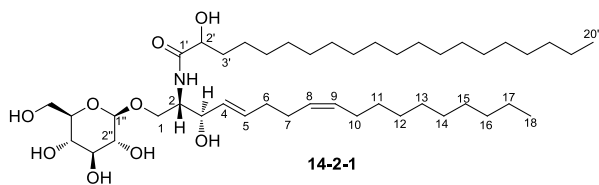
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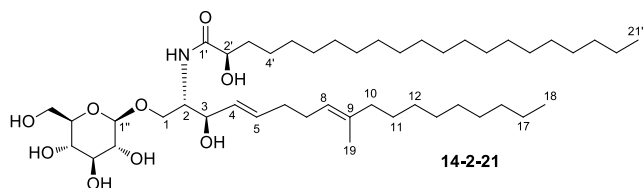
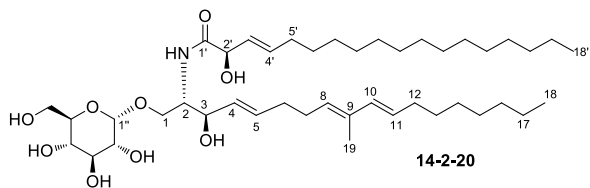
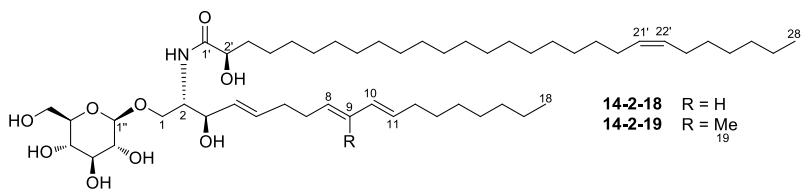
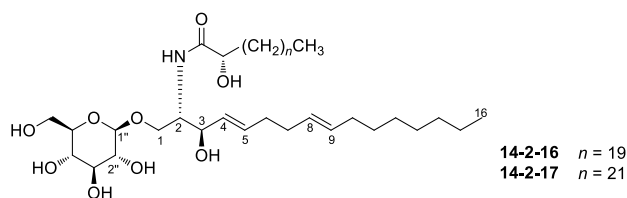
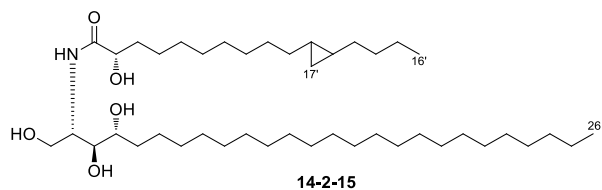
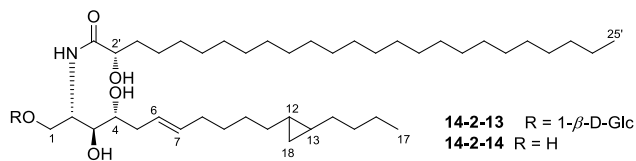
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14.2 Cerebrosides

Table 14-2-1: Compounds, MFs, and test solvents of cerebroside 14-2-1~14-2-29.

No.	Compounds	MFs	Test solvents	Refer-ences
14-2-1	1- <i>O</i> - β -D-glucopyranosyl-(2 <i>S</i> ,3 <i>R</i> ,4 <i>E</i> ,8 <i>Z</i>)-2-[(2 <i>R</i>)-hydroxyicosanoyl]amido]-4,8-octadecadiene-1,3-diol	C ₄₄ H ₈₃ NO ₉	CD ₃ OD	[12]
14-2-2	1- <i>O</i> - β -D-glucopyranosyl-(2 <i>S</i> ,3 <i>R</i> ,4 <i>E</i> ,8 <i>E</i>)-2-[(2-hydroxyicosanoyl]amido]-4,8-octadecadiene-1,3-diol	C ₄₄ H ₈₃ NO ₉	CD ₃ OD	[12]
14-2-3	1- <i>O</i> - β -D-glucopyranosyl-(2 <i>S</i> ,3 <i>R</i> ,4 <i>E</i> ,8 <i>Z</i>)-2- <i>N</i> -palmitoyl-octadecasphinga-4,8-dienine	C ₄₀ H ₇₅ NO ₈	CDCl ₃ - CD ₃ OD(2:1)	[13]
14-2-4	1- <i>O</i> - β -D-glucopyranosyl-(2 <i>S</i> ,3 <i>R</i> ,4 <i>E</i> ,8 <i>Z</i>)-2- <i>N</i> -(2'-hydroxypalmitoyl)octadecasphinga-4,8-dienine	C ₄₀ H ₇₅ NO ₉	CDCl ₃ - CD ₃ OD(2:1)	[13]
14-2-5	lactariamide A	C ₄₂ H ₈₃ NO ₄	C ₅ D ₅ N	[14]
14-2-6	lactariamide B	C ₃₇ H ₇₁ NO ₄	CDCl ₃	[14]
14-2-7	turbostatin 1	C ₃₈ H ₇₃ NO ₈	C ₅ D ₅ N	[15]
14-2-8	turbostatin 2	C ₄₀ H ₇₇ NO ₈	C ₅ D ₅ N	[15]
14-2-9	turbostatin 3	C ₃₈ H ₇₃ NO ₈	C ₅ D ₅ N	[15]
14-2-10	turbostatin 4	C ₄₀ H ₇₇ NO ₈	C ₅ D ₅ N	[15]
14-2-11	asteriaceramide A	C ₃₈ H ₇₅ NO ₅	C ₅ D ₅ N	[16]
14-2-12	asteriacerebroside G	C ₄₂ H ₇₉ NO ₉	C ₅ D ₅ N	[16]
14-2-13	gracilarioside	C ₄₉ H ₉₃ NO ₁₀	C ₅ D ₅ N	[17]
14-2-14	gracilamide A	C ₄₃ H ₈₃ NO ₅	C ₅ D ₅ N	[17]
14-2-15	gracilamide B	C ₄₃ H ₈₅ NO ₅	C ₅ D ₅ N	[17]
14-2-16	ferocerebroside A	C ₄₄ H ₈₃ NO ₉	CDCl ₃ - CD ₃ OD(1:1)	[18]
14-2-17	ferocerebroside B	C ₄₆ H ₈₇ NO ₉	CDCl ₃ - CD ₃ OD(1:1)	[18]
14-2-18	renieroside A ₁	C ₅₂ H ₉₅ NO ₉	CD ₃ OD	[19]
14-2-19	renieroside B ₁	C ₅₃ H ₉₇ NO ₉	CD ₃ OD	[19]
14-2-20	sarcoehrenoside A	C ₄₃ H ₇₇ NO ₉	CD ₃ OD	[20]
14-2-21	sarcoehrenoside B	C ₄₆ H ₈₇ NO ₉	CD ₃ OD	[20]
14-2-22	alternaroside A	C ₄₃ H ₇₇ NO ₁₀	DMSO- <i>d</i> ₆	[21]
14-2-23	alternaroside B	C ₄₂ H ₇₇ NO ₉	DMSO- <i>d</i> ₆	[21]
14-2-24	alternaroside C	C ₄₂ H ₇₇ NO ₉	DMSO- <i>d</i> ₆	[21]
14-2-25	catocerebroside A	C ₄₉ H ₉₁ NO ₉	CD ₃ OD	[22]
14-2-26	catocerebroside B	C ₄₈ H ₉₃ NO ₁₀	C ₅ D ₅ N	[22]
14-2-27	catocerebroside C	C ₄₈ H ₉₃ NO ₁₁	C ₅ D ₅ N	[22]
14-2-28	temnoside A	C ₄₉ H ₉₇ NO ₉	C ₅ D ₅ N	[23]
14-2-29	temnoside B	C ₄₈ H ₉₅ NO ₈	C ₅ D ₅ N	[23]





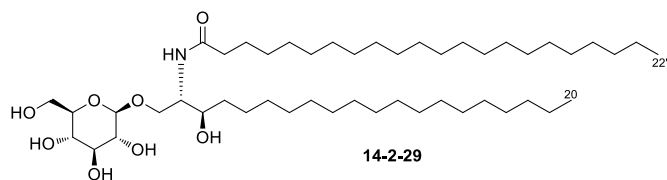
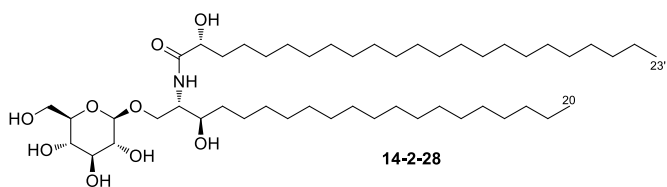
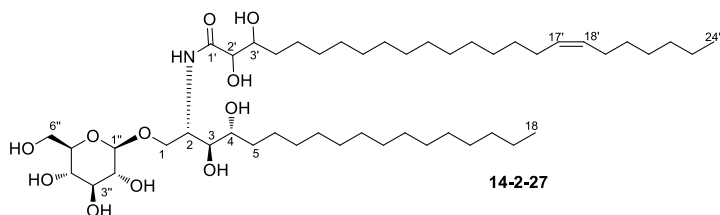
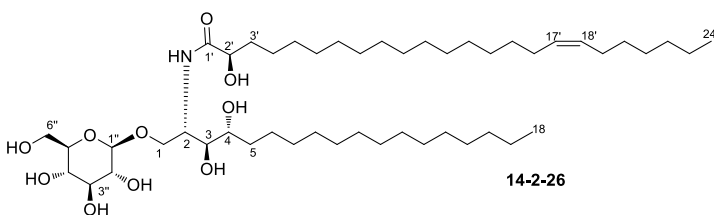
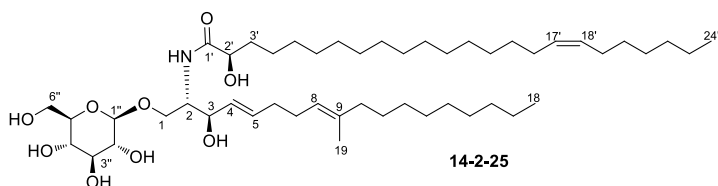
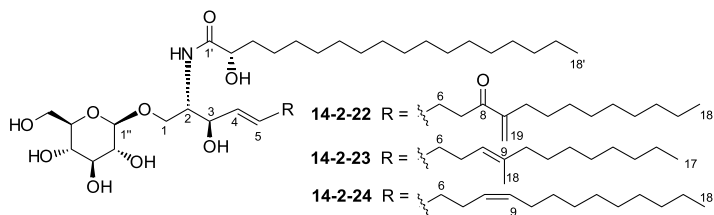


Table 14-2-2: ^1H NMR spectroscopic data of cerebrosides 14-2-1~14-2-4.

H	14-2-1	14-2-2	14-2-3	14-2-4
1	4.11 dd(10.1, 5.8) 3.71 dd(10.1, 3.7)	4.10 dd(10.1, 5.2) 3.70 dd(10.1, 3.8)	4.07	4.07
2	3.99 ddd(5.8, 3.7, 1.9)	3.98 ddd(5.8, 3.7, 2.0)	4.02	4.02
3	4.14 td(7.5, 1.9)	4.13 td(8.3, 1.5)	4.12	4.12
4	5.49 dd(15.1, 7.5)	5.47 dd(15.5, 7.6)	5.48 dd(16)	5.48 dt(16.0)
5	5.73 dtd(15.1, 6.2, 1.5)	5.72 br d(15.5)	5.75 dt(16)	5.75 dt(16.0)
6	2.07 m	2.07 m	2.05 m	2.10 m
7	2.12 m	2.07 m	1.98 m	2.01 m
8	5.37 t(5.1)	5.42 t(4.9)	5.35 t(8.0)	5.35 t(8.0)
9	5.37 t(5.1)	5.42 t(4.9)	5.35 t(8.0)	5.35 t(8.0)
10	2.05 m	1.98 m	1.98 m	1.98 m
11	1.35 m	1.35 m	1.37 m	1.37 m
12~17	1.30 m	1.30 m	1.27 m	1.30 m
18	0.90 t(7.2)	0.90 t(7.2)	0.89 t(6.9)	0.90 t(6.9)
1''	4.26 d(8.0)	4.26 d(7.9)	4.27 d(7.8)	4.26 d(8.0)
2''	3.19 dd(9.0, 8.0)	3.18 dd(9.0, 7.9)	3.19	3.25
3''	3.35 t(9.0)	3.34 t(9.0)	3.24 t(8.8)	3.38 t(9.0)
4''	3.28 m	3.26 m	3.28	3.35
5''	3.27 m	3.27 m	3.24 t(8.8)	3.30 t(9.0)
6''	3.86 dd(12.0, 2.8) 3.67 dd(12.0, 5.8)	3.86 dd(12.1, 1.7) 3.66 dd(12.1, 5.6)	3.86	3.86
2'	3.98 dd(8.0, 4.2)	3.97 dd(8.0, 3.8)	2.15	3.99
3'	1.72 m, 1.54 m	1.70 m, 1.55 m	1.75	1.75
4'	1.40 m	1.40 m	1.42 m	1.55 m
5'~19'	1.30 m	1.30 m		
5'~16'			1.27 m	1.30 m
18'			0.89 t(6.9)	0.90 t(6.9)
20'	0.90 t(7.0)	0.90 t(6.9)		

Table 14-2-3: ^1H NMR spectroscopic data of cerebrosides 14-2-5 and 14-2-6.

H	14-2-5	14-2-6	H	14-2-5	14-2-6
1	4.50 dd(10.8, 5.0) 4.60 dd(10.8, 4.5)	3.72 m~3.78 m	18	0.90 t(7.2)	0.90 t(7.3)
2	5.20 m	4.22 m	19		1.60 s
3	4.45 dd(6.5, 4.7)	4.08 dd(7.2, 4.4)	NH	8.65 d(9.9)	7.21 d(7.8)
4	4.35 td(7.7, 2.3)	5.10 m	2'	4.75 dd(7.5, 3.8)	3.90 dd(10.8, 4.0)
5	2.35 m, 2.08 m	5.78 m	3'	2.18 m, 2.28 m	1.55 m, 1.75 m
6	1.80 m, 1.51 m	2.05 m	4'	1.49 m, 1.90 m	1.39 m
7		2.05 m	5'~15'		1.22~1.32 m
8		5.50 dd(15.6, 6.4)	5'~21'	1.25~1.45 m	
10		1.95 t(6.8)	16'		1.22~1.32 m
			17'		1.22~1.32 m

Table 14-2-3 (continued)

H	14-2-5	14-2-6	H	14-2-5	14-2-6
7~15	1.33~1.37 m		22'	1.25~1.45 m	
11~15		1.22~1.32 m	23'	1.25~1.45 m	
16	1.25~1.45 m	1.22~1.32 m	Me	0.90 t(7.2)	0.90 t(7.3)
17	1.25~1.45 m	1.22~1.32 m			

Table 14-2-4: ¹H NMR spectroscopic data of cerebrosides 14-2-7~14-2-10.

H	14-2-7	14-2-8	14-2-9	14-2-10
1	4.82 m, 4.23 m	4.82 m, 4.22 m	4.81 m, 4.24 m	4.82 m, 4.23 m
2	4.81 m	4.80 m	4.80 m	4.81 m
3	4.75 m	4.75 m	4.75 m	4.75 m
4	5.98 dd(6.0, 15.0)	5.97 dd(6.0, 15.2)	5.99 dd(6.0, 15.2)	5.98 dd(6.0, 15.0)
5	5.85 dt(6.0, 15.0)	5.83 dt(6.0, 15.2)	5.85 dt(6.0, 15.2)	5.83 dt(6.0, 15.0)
6	2.03 q(7.0)	2.01 q(7.1)	2.03 q(7.2)	2.01 q(7.0)
7~15	1.21 br s	1.21 br s	1.21 br s	1.21 br s
16	0.85 t(8.6)	0.85 t(8.6)	0.85 t(8.6)	0.85 t(8.6)
NH	8.35 d(7.5)	8.37 d(7.8)	8.37 d(7.7)	8.36 d(7.8)
1''	4.96 d(8.2)	4.96 d(8.1)	4.89 d(7.5)	4.88 d(7.3)
2''	4.06 dd(8.4, 9.6)	4.04 dd(8.1, 9.3)	4.52 dd(7.5, 9.5)	4.52 dd(7.5, 9.4)
3''	4.24 m	4.24 m	4.16 dd(3.0, 9.5)	4.14 dd(3.0, 9.4)
4''	4.37 m	4.37 m	4.56 d(3.0)	4.55 d(3.0)
5''	3.90 m	3.91 m	4.07 dd(6.0, 9.0)	3.07 dd(6.2, 8.9)
6''	4.51 dd(5.1, 11.7)	4.50 dd(5.0, 11.6)	4.44 m	4.44 m
	4.20 dd(2.3, 11.7)	4.22 dd(2.3, 11.6)		
2'	2.41 t(7.0)	2.41 t(7.0)	2.41 t(7.2)	2.41 t(7.1)
3'	1.80 m	1.80 m	1.80 m	1.80 m
4'~17'		1.21 br s		1.21 br s
4'~15'	1.21 br s		1.21 br s	
18' or 16'	0.85 t(8.6)	0.85 t(8.6)	0.85 t(8.6)	0.85 t(8.6)

Table 14-2-5: ¹H NMR spectroscopic data of cerebrosides 14-2-11 and 14-2-12.

H	14-2-11	14-2-12	H	14-2-11	14-2-12
1	4.52 dd(10.9, 4.8)	4.25 m	Me	0.86 m(6H)	0.86 m(6)
	4.43 dd(10.9, 4.8)	4.72 m	1''		4.92 d(8.0)
2	5.13 m	4.80 m	2''		4.04 dd(8.3, 8.0)
3	4.36 m	4.80 m	3''		4.21 m
4	4.29 m	5.98 m	4''		4.23 m
5		5.90 m	5''		3.91 m
13	5.49 m	5.50 t	6''		4.36 dd(11.7, 5.4), 4.51 m
2'	4.63 dd(7.6, 3.7)	4.58 m	NH	8.59 d(8.8)	8.36 d(8.7)

Table 14-2-6: ^1H NMR spectroscopic data of cerebrosides 14-2-13~14-2-15.

H	14-2-13	14-2-14	H	14-2-15
1	4.74 dd(10.6, 6.4) 4.48 dd(10.6, 4.2)	4.62 dd(10.5, 4.0) 4.50 dd(10.5, 6.2)	1	4.52 dd(10.7, 4.6) 4.43 dd(10.7, 5.0)
2	5.24 m	5.10 m	2	5.14 m
3	4.30 m	4.33 m	3	4.35 m
4	4.23 m	4.30 m	4	4.28 m
5	2.99 m, 2.67 m	3.06 m, 2.72 m	5	1.93 m, 2.24 m
6	5.95 ddd(14.8, 8.0, 6.9)	5.98 ddd(14.5, 8.1, 6.8)	6	1.91 m
7	5.72 ddd(14.8, 7.6, 6.6)	5.73 ddd(14.5, 8.3, 7.1)	7~23,5'~9'	1.26
8	2.05 m	2.04 m	24,14'	1.26
9~10	1.26	1.26	25,15'	1.26
4'~22'	1.46	1.44	26,16'	0.86
11,14	1.46	1.44	1'	
12,13	0.74 m	0.72 m	2'	4.62 m
15,23'	1.26	1.26	3'	2.05 m, 2.21 m
16,24'	1.26	1.26	4'	1.69 m, 1.78 m
17,25'	0.88 t(6.8)	0.85	10',13'	1.43
18	-0.21 ddd(5.3, 5.3, 4.3) 0.66 ddd(4.3, 3.9, 3.9)	-0.20 ddd(5.1, 5.1, 3.8) 0.65 ddd(3.8, 3.3, 3.3)	11',12'	0.73 m
2'	4.54 m	4.53 m	17'	-0.22 ddd(5.0, 5.0, 4.5) 0.68 ddd(4.5, 3.5, 3.5)
3'	2.17 m, 1.96 m	2.22 m, 1.94 m		
1''	4.86 d(7.7)			
2''	4.41 t(7.8)			
3''	4.10 m			
4''	4.50 m			
5''	3.98 m			
6''	4.43 dd(12.6, 5.0) 4.40 dd(12.6, 5.0)			
NH	8.50	8.58		
2'	4.54 m	4.53 m		8.60

Table 14-2-7: ^1H NMR spectroscopic data of cerebrosides 14-2-16 and 14-2-17.

H	14-2-16	14-2-17	H	14-2-16	14-2-17
1	4.09 dd(10.2, 5.5) 3.73 m	4.10 dd(10.2, 5.5) 3.75 m	1''	4.27 d(7.8)	4.29 d(8.0)
2	4.00 m	4.02 m	2''	3.24 dd(8.9, 7.8)	3.34 dd(9.0, 8.0)
3	4.13 t(7.2)	4.15 t(7.4)	3''	3.40 t(8.9)	3.45 t(9.0)
4	5.48 dd(15.5, 7.2)	5.50 dd(15.6, 7.4)	4''	3.38 t(8.9)	3.39 t(9.0)
5	5.74 dt(15.5, 6.4)	5.78 dt(15.6, 6.4)	5''	3.28 m	3.30 m
6	2.08 m(2H)	2.05 m(2H)	6''	3.71 m	3.75 dd(12.2, 5.2)
7	2.08 m(2H)	2.05 m(2H)		3.87 dd(12.1, 2.5)	3.87 dd(12.2, 2.5)
8	5.41 m	5.40 m	2'	4.02 dd(7.5, 3.8)	4.05 dd(7.8, 3.8)
9	5.41 m	5.40 m	3'	1.70 m, 1.45 m	1.70 m, 1.45 m
10	1.95 m(2H)	1.95 m(2H)	Me	0.88 t(7.0)	0.87 t(6.5)
Me	0.88 t(7.0)	0.87 t(7.0)	NH	8.33 d(8.6)	8.35 d(8.5)

Table 14-2-8: ^1H NMR spectroscopic data of cerebrosides **14-2-18~14-2-21**.

H	14-2-18	14-2-19	H	14-2-20	14-2-21
1	4.13 m 3.68 m	4.13 m 3.67 m	1	3.88 dd(10.5, 4.5) 3.83 dd(10.5, 4.5)	4.12 dd(10.5, 3.5) 3.71 dd(10.5, 3.5)
2	3.97 m	3.98 m	2	3.93 dd(8.0, 4.5)	3.99 dd(7.5, 3.5)
3	4.12 m	4.13 m	3	4.14 t(8.0)	4.13 t(7.5)
4	5.48 dd(15.0, 7.5)	5.48 dd(15.0, 8.0)	4	5.47 dd(15.5, 8.0)	5.48 dd(15.5, 7.5)
5	5.73 dt(15.0, 5.5)	5.73 dt(15.0, 6.5)	5	5.72 dd(15.5, 6.5)	5.74 dd(15.5, 6.0)
6	2.14 m	2.09 m	6	2.08 td(7.5, 6.5)	2.06 m
7	2.09 m	2.21 m	7	2.20 td(7.5, 7.0)	2.07 m
8	5.56 m	5.33 m	8	5.35 t(7.0)	5.14 t(6.5)
9	5.99 m		9		
10	5.99 m	6.02 d(15.5)	10	6.02 d(15.5)	1.97 t(7.5)
11	5.54 m	5.52 dt(15.0, 7.0)	11	5.55 dt(15.5, 7.0)	1.39 m
12	2.06 m	2.07 q(7.0)	12	2.08 td(7.5, 7.0)	
13~16	1.28~1.24 m	1.38~1.28 m	13	1.38 m	
17	1.38~1.24 m	1.38~1.28 m	12~15		1.29 m
18	0.89 t(7.0)	0.89 t(7.0)	14,15	1.29 m	
19		1.71 s	16	1.29 m	1.29 m
2'	4.12 dd(8.0, 4.8)	4.12 dd(8.0, 4.8)	17	1.29 m	1.29 m
3'	1.71 m, 1.56 m	1.69 m, 1.54 m	18	0.90 t(6.0)	0.90 t(6.5)
4'~19' or 24'~26'	1.38~1.24 m	1.38~1.24 m	19	1.71 s	1.59 s
20' or 23'	2.02 q(6.0)	2.02 q(6.0)	1'		
21' or 22'	5.34 m	5.34 m	2'	4.45 d(6.0)	3.99 dd(8.0, 4.0)
27'	1.28~1.24 m	1.38~1.24 m	3'	5.50 dd(15.5, 6.0)	1.70 m, 1.55 m
28'	0.89 t(7.0)	0.89 t(7.0)	4'	5.83 dd(15.5, 6.5)	1.41 m
1''	4.26 d(7.5)	4.26 d(7.5)	5'	2.03 td(7.5, 6.5)	
2''	3.18 m	3.18 m	5'~19'		1.29 m
3''	3.35 m	3.35 m	6'	1.38 m	
4''	3.28 m	3.28 m	7'~15'	1.29 m	
5''	3.28 m	3.28 m	16'	1.29 m	
6''	3.87 dd(12.0, 1.0) 3.71 m	3.87 dd(12.0, 1.0) 3.71 m	17'	1.29 m	
			18'	0.90 t(6.0)	
			20'		1.29 m
			21'		0.90 t(6.5)
			1''	4.87 d(4.5)	4.27 d(8.0)
			2''	3.96 dd(7.5, 4.5)	3.19 dd(9.5, 8.0)
			3''	4.10 t(7.5)	3.34 m
			4''	3.63 m	3.30 m
			5''	3.69 m	3.28 m
			6''	3.63 m, 3.56 m	3.87 dd(12.0, 1.5) 3.67 dd(12.0, 5.0)

Table 14-2-9: ^1H NMR spectroscopic data of cerebrosides 14-2-22~14-2-24.

H	14-2-22	14-2-23	14-2-24
1	3.51 dd(10.2, 4.1), 3.94 m	3.51 dd(10.6, 4.1), 3.94 m	3.51 dd(10.1, 4.1), 3.95 m
2	3.78 m	3.78 m	3.78 m
3	3.98 m	3.97 m	3.97 m
4	5.40 dd(15.6, 6.9)	5.37 dd(15.6, 6.9)	5.36 dd(15.6, 6.9)
5	5.58 dt(15.6, 6.4)	5.57 dt(15.6, 6.2)	5.56 dt(14.2, 6.2)
6	2.16 m	1.94 m	1.97 m
7	2.73 t(7.6)	1.94 m	1.97 m
8		5.09 br t(6.2)	5.38 t(3.3)
9			5.38 t(3.3)
10	2.15 m	1.94 m	1.97 m
11	1.23 m	1.23 m	1.23 m
12~14	1.23 m	1.23 m	1.23 m
15	1.23 m	1.23 m	1.23 m
16	1.23 m	1.23 m	1.23 m
17	1.23 m	0.85 t(6.9)	1.23 m
18	0.85 t(6.9)	1.54 br s	0.85 t(6.8)
19	6.09 br s, 5.79 br s		
2'	4.30 br t(5.1)	4.30 br t(4.6)	4.30 br t(5.1)
3'	5.44 dd(15.1, 5.1)	5.44 dd(15.4, 5.3)	5.43 dd(15.1, 5.5)
4'	5.67 dt(15.1, 6.9)	5.68 dt(15.4, 6.4)	5.68 dt(15.1, 6.9)
5'	1.93 dt(6.9, 6.8)	1.94 m	1.97 m
6'~15'	1.23 m	1.23 m	1.23 m
16'	1.23 m	1.23 m	1.23 m
17'	1.23 m	1.23 m	1.23 m
18'	0.85 t(6.6)	0.85 t(6.9)	0.85 t(6.8)
1''	4.12 d(7.8)	4.11 d(7.8)	4.12 d(7.8)
2''	2.96 m	2.96 m	2.96 m
3''	3.14 m	3.14 m	3.14 m
4''	3.05 m	3.04 m	3.04 m
5''	3.09 m	3.08 m	3.09 m
6''	3.67 br dd(11.0, 6.0)	3.66 br dd(9.7, 6.0)	3.67 br dd(11.3, 5.9, 1.9)
	3.46 ddd(11.0, 6.0, 5.0)	3.43 ddd(9.7, 6.0, 5.5)	3.43 ddd(11.3, 5.9 5.0)
NH	7.39 d(9.1)	7.39 d(9.7)	7.39 d(9.6)
3-OH	4.97 m	4.95 d(5.5)	4.95 d(5.5)
2'-OH	5.78 d(5.0)	5.78 d(5.0)	5.78 d(5.4.6)
2''-OH	4.99 m	4.98 d(4.6)	4.99 d(4.6)
3''-OH	4.93 m	4.92 d(5.0)	4.92 d(5.0)
4''-OH	4.94 m	4.94 d(4.6)	4.93 d(5.0)
6''-OH	4.53 t(6.0)	4.53 t(6.0)	4.52 t(5.9)

Table 14-2-10: ^1H NMR spectroscopic data of cerebrosides 14-2-25~14-2-27.

H	14-2-25	H	14-2-26	14-2-27
1	4.30 dd(10.5, 3.5) 3.90 dd(10.5, 7.0)	1	4.75 dd(10.7, 6.6) 4.50 dd(10.7, 4.5)	4.70 dd(10.6, 6.0) 4.50 dd(10.6, 4.6)
2	4.19 m	2	5.26 m	5.28 m
3	4.31 t(6.9)	3	4.34 m	4.29 m
4	5.68 dd(15.3, 7.4)	4	4.22 m	4.23 m
5	5.93 dt(15.3, 5.8)	5	2.11 m, 1.97 m	2.23 m, 1.93 m
6	2.25 m	6	1.80 m(2H)	1.85 m
7	2.23 m	7~17, 5'~15'(6'~15'), 20'~23'	1.26~1.38 m	1.25~1.457
8	5.33 m	18, 24'	0.93 t(6H, 7.6)	0.87 t(6H, 6.8)
10	2.17 t(7.6)	NH	8.55 d(9.1)	8.66 d(9.2)
11	1.48 m	2'	4.60 m	4.70 m
12~15	1.40~1.55	3'	2.10 m, 2.00 m	4.51 m
16	1.40~1.55	4'	1.85 m	2.13 m
17	1.40~1.55	5'		1.85 m
18	1.07 t(7.2)	16', 19'	2.07 m	2.13 m
19	1.79 s	17', 18'	5.50 m	5.51 m
		1''	4.94 d(7.7)	4.97 d(7.7)
2'	4.18 m	2''	4.01 t(8.0)	3.98 m
3'	1.70 m, 1.80 m	3''	4.19 m	4.17 m
4'	1.52 m	4''	4.20 m	4.19 m
5'~15'	1.40~1.50	5''	3.87 m	3.84 m
16'	2.24 m	6''	4.32 dd(11.6, 5.5)	4.52 d(11.7)
17', 18'	5.53 m		4.52 d(11.6)	4.31 dd(11.7, 5.4)
19'	2.24 m			
20'~21'	1.40~1.50			
22'	1.40~1.50			
23'	1.40~1.50			
24'	1.07 t(7.2)			
1''	4.46 d(8.0)			
2''	3.38 t(7.8)			
3''	3.49 m			
4''	3.50 m			
5''	3.51 m			
6''	4.06 d(11.7), 3.86 dd(11.7, 5.2)			

Table 14-2-11: ^1H NMR spectroscopic data of cerebrosides **14-2-28** and **14-2-29**.

H	14-2-28	14-2-29	H	14-2-28	14-2-29
1	4.75 dd(6.1, 12.5) 4.35 dd(6.0, 12.8)	4.35 dd(6.1, 12.9) 4.75 dd(5.7, 12.8)	4' $\sim$ 22' 4' $\sim$ 21'	1.21 br s	1.24 br s
2	5.29 m	5.29 m	22'		0.85 t(7.8)
3	4.65 m	4.66 m	23'	0.85 t(8.7)	
4	1.95 m	1.94 m	1''	4.95 d(8.1)	4.95 d(8.4)
5~19	1.21 br s	1.24 br s	2''	4.05 dd(8.4, 9.7)	4.05 t(8.4)
20	0.85 t(8.7)	0.85 t(7.8)	3''	4.25 m	4.25 m
NH	8.60 d(9.1)	8.52 d(9.0)	4''	4.36 m	4.36 m
2'	4.28 m	2.42 t(7.2)	5''	3.90 m	3.90 m
3'	1.82 m	1.77~1.85 m	6''	4.52 dd(5.2, 11.6) 4.20 dd(2.2, 11.8)	4.20 m, 4.55 m

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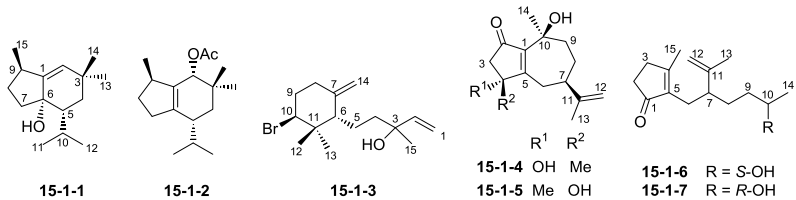
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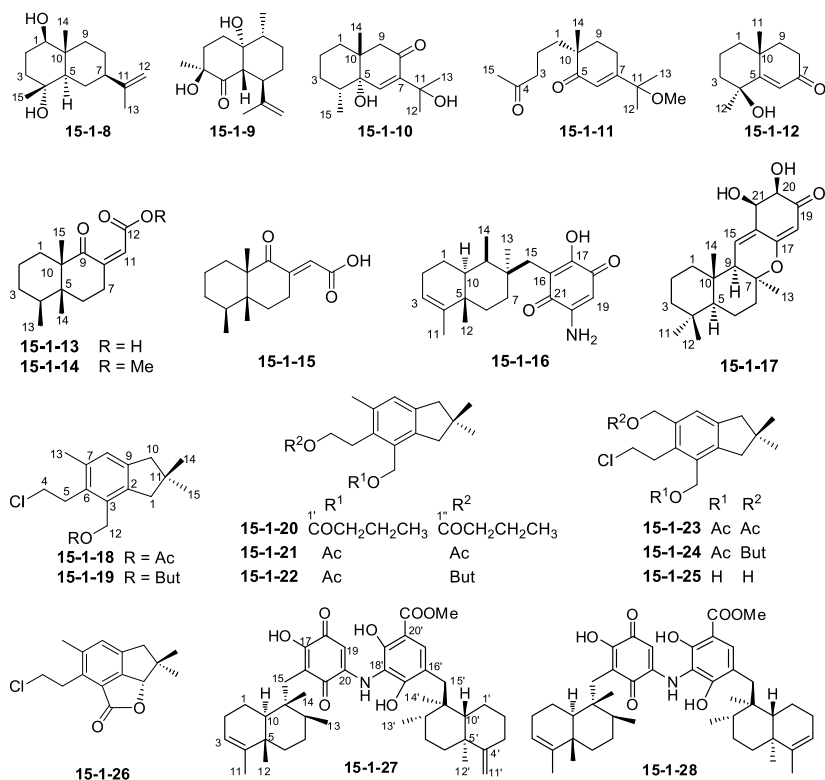
15.1 Marine terpenoids

Table 15-1-1: Compounds, MFs, and test solvents of marine terpenoids 15-1-1~15-1-28.

No.	Compounds	MFs	Test solvents	References
15-1-1	6-hydroxy-1-brasilene	C ₁₅ H ₂₆ O	CDCl ₃	[1]
15-1-2	epibrasilenol acetate	C ₁₇ H ₂₈ O ₂	CDCl ₃	[1]
15-1-3	6- <i>epi</i> - β -snyderol	C ₁₅ H ₂₅ OB _r	CDCl ₃	[1]
15-1-4	cyperusol A ₁	C ₁₅ H ₂₂ O ₃	C ₅ D ₅ N	[2]
			CDCl ₃	[2]
15-1-5	cyperusol A ₂	C ₁₅ H ₂₂ O ₃	C ₅ D ₅ N	[2]
			CDCl ₃	[2]
15-1-6	cyperusol B ₁	C ₁₅ H ₂₄ O ₂	CDCl ₃	[2]
15-1-7	cyperusol B ₂	C ₁₅ H ₂₄ O ₂	CDCl ₃	[2]
15-1-8	cyperusol C	C ₁₅ H ₂₆ O ₂	CDCl ₃	[2]
15-1-9	cyperusol D	C ₁₅ H ₂₄ O ₃	C ₅ D ₅ N	[2]
			CDCl ₃	[2]
15-1-10	(4 <i>R</i> *,5 <i>S</i> *,6 <i>Z</i> ,10 <i>R</i> *)-8-oxo-eudesm-6-en-5 α ,11-diol	C ₁₅ H ₂₄ O ₃	CDCl ₃	[3]
			C ₅ D ₅ N	[3]
15-1-11	(6 <i>E</i> ,10 <i>R</i>)-4,5-dioxo-11-methoxy-eudesm-6-ene	C ₁₆ H ₂₆ O ₃	CDCl ₃	[3]
15-1-12	(4 <i>S</i> *,5 <i>E</i> ,10 <i>R</i> *)-7-oxo-tri- <i>nor</i> -eudesm-5-en-4 β -ol	C ₁₂ H ₁₈ O ₂	CDCl ₃	[3]
15-1-13	aignopsanoic acid A	C ₁₅ H ₂₂ O ₃	CDCl ₃	[4]
15-1-14	(4 <i>S</i> ,5 <i>R</i> ,10 <i>S</i>)-methyl aignopsanoate A	C ₁₆ H ₂₄ O ₃	CDCl ₃	[4]
15-1-15	isoaignopsanoic acid A	C ₁₅ H ₂₂ O ₃	CDCl ₃	[4]
15-1-16	dysideamine	C ₂₁ H ₂₉ NO ₃	CDCl ₃	[5]
15-1-17	puupehanol	C ₂₁ H ₃₀ O ₄	CDCl ₃	[6]
15-1-18	12-acetylalcyopterosin D	C ₁₇ H ₂₃ O ₂ Cl	CDCl ₃	[7]
15-1-19	12- <i>n</i> -butanoylalcyopterosin D	C ₁₉ H ₂₇ O ₂ Cl	CDCl ₃	[7]
15-1-20	4,12-bis- <i>n</i> -butanoylalcyopterosin O	C ₂₃ H ₃₄ O ₄	CDCl ₃	[7]
15-1-21	4,12-bis(acetyl)alcyopterosin O	C ₁₉ H ₂₆ O ₄	CDCl ₃	[7]
15-1-22	12-acetyl-4- <i>n</i> -butanoylalcyopterosin O	C ₂₁ H ₃₀ O ₄	CDCl ₃	[7]
15-1-23	13-acetoxy-12-acetylalcyopterosin D	C ₁₉ H ₂₅ O ₄ Cl	CDCl ₃	[7]
15-1-24	12-acetyl-13- <i>n</i> -butanoxyalcyopterosin D	C ₂₁ H ₂₉ O ₄ Cl	CDCl ₃	[7]
15-1-25	13-hydroxyalcyopterosin D	C ₁₅ H ₂₁ O ₂ Cl	CDCl ₃	[7]
15-1-26	alcyopterosin P	C ₁₅ H ₁₇ O ₂ Cl	CDCl ₃	[7]
15-1-27	nakijiquinone E	C ₄₄ H ₅₉ O ₇ N	CDCl ₃	[8]
15-1-28	nakijiquinone F	C ₄₄ H ₅₉ O ₇ N	CDCl ₃	[8]



<https://doi.org/10.1515/9783110631616-015>

**Table 15-1-2:** ¹H NMR spectroscopic data of marine terpenoids 15-1-1~15-1-3 and 15-1-6.

H	15-1-1	15-1-2	15-1-3	15-1-6
1			5.17 br d(17.4), 5.04 br d(10.7)	
2	5.05 br s	4.96 br s	5.84 dd(17.4, 10.7)	2.34 m
3				2.48 m
4	1.38 m, 1.33 m	1.41 m, 1.09 m	1.39 m, 1.27 m	
5	1.24 m	2.05 m	1.58 m, 1.28 m	
6			1.95 m	2.23 br d(ca. 7)
7	α 1.50 m, β 1.95 m	2.27 m, 1.59 m		2.31 m
8	2.12 m, 1.18 m	2.08 m, 1.38 m	2.11 m	1.37 m
9	2.68 m	2.60 m	α 2.14 m, β 1.98 m	1.45 m
10	1.97 m	1.98 m	4.31 dd(11.2, 4.0)	3.76 m
11	0.96 d(6.9)	0.70 d(6.8)		
12	0.97 d(6.8)	0.91 d(6.8)	1.01 s	4.58 br s, 4.68 m
13	1.02 s	0.82 s	1.04 s	1.63 br s
14	0.89 s	0.90 s	4.78 br s, 4.59 br s	1.16 d(6.1)
15	0.99 d(6.8)	0.97 d(6.9)	1.25 s	
OAc		2.01 s		

Table 15-1-3: ^1H NMR spectroscopic data of marine terpenoids **15-1-4** and **15-1-5**.

H	15-1-4 ($\text{C}_5\text{D}_5\text{N}$)	15-1-4 (CDCl_3)	15-1-5 ($\text{C}_5\text{D}_5\text{N}$)	15-1-5 (CDCl_3)
3	2.98 d(18.0), 2.77 d(18.0)	2.61 s	2.93 d(18.0), 2.84 d(18.0)	2.60 s
6 α	3.01 br d(17)	2.64 br d(ca. 17)	2.78 dd(2.8, 16.4)	2.52 dd(3.1, 16.5)
6 β	2.68 dd(11.3, 16.5)	2.50 dd(11.0, 16.8)	2.98 dd(10.0, 16.4)	2.63 dd(9.2, 16.5)
7	2.41 m	2.39 m	2.50 m	2.39 m
8	1.82 m, 1.74 m	1.87 m, 1.67 m	1.88 m	1.81 m
9 α	1.89 ddd(4.3, 4.6, 13.4)	1.72 m	1.85 m	1.73 ddd(4.3, 4.6, 14.0)
9 β	2.31 ddd(6.1, 12.8, 13.4)	2.07 ddd(5.8, 13.7, 13.8)	2.27 ddd(6.1, 12.5, 13.2)	2.07 ddd(5.8, 13.4, 14.0)
12	4.75 br s, 4.77 br s	4.75 m	4.80 br s, 4.10 br s	4.78 m
13	1.69 br s	1.76 br s	1.72 s ^①	1.77 s ^①
14	1.47 s	1.46 s	1.70 s ^①	1.47 s ^①
15	1.59 s	1.49 s	1.57 s ^①	1.47 s ^①

^①The chemical shifts of 13-, 14-, and 15-Me were not assigned in the literature, and here, the placements are only to display the data.

Table 15-1-4: ^1H NMR spectroscopic data of marine terpenoids **15-1-7~15-1-9**.

H	15-1-7	15-1-8	15-1-9 ($\text{C}_5\text{D}_5\text{N}$)	15-1-9 (CDCl_3)
1		3.32 dd(4.4, 11.2)		
2	2.34 m	1.72 m 1.62 m	α 2.11 m β 2.38 ddd(4.0, 13.0, 13.5)	1.84 m
3	2.49 m	α 1.52 ddd(3.5, 12.0, 13.5) β 1.79 ddd(3.0, 3.5, 12.0)	2.18 m 2.33 m	1.92 m
5		1.28 m		
6	2.23 br d(ca. 7)	1.84 m, 1.26 m	3.95 d(12.0)	3.48 d(12.0)
7	2.27 m	1.94 m	3.03 ddd(4.0, 12.0, 13.0)	2.35 ddd(3.5, 12.0, 13.0)
8	1.35 m	α 1.61 m β 1.38 dddd (3.5, 13.0, 13.5, 17.0)	α 1.93 dddd(4.0, 4.0, 6.5, 16.5) β 1.33 dddd (4.0, 13.0, 13.0, 16.5)	α 1.79 m β 1.18 dddd (3.5, 13.0, 13.0, 16.5)
9	1.47 m	α 1.13 ddd (4.0, 13.0, 13.5) β 1.90 ddd (3.5, 3.5, 13.5)	α 1.80 dddd (4.0, 13.0, 13.0, 16.5) β 1.44 dddd (4.0, 6.5, 6.5, 16.5)	α 1.34 dddd (3.5, 13.0, 13.5, 16.5) β 1.49 dddd (3.5, 7.0, 7.0, 16.5)
10	3.75 m		1.72 m	1.67 m
12	4.58 br s, 4.68 m	4.72 m	4.73 m, 4.92 br s	4.60 m, 4.66 br s

Table 15-1-4 (continued)

H	15-1-7	15-1-8	15-1-9 (C ₅ D ₅ N)	15-1-9 (CDCl ₃)
13	1.63 br s	0.89 s ^①	1.90 s ^①	1.79 s ^①
14	1.17 d(6.1)	1.11 s ^①	1.10 d(6.4)	0.92 d(6.4)
15		1.75 s ^①	1.56 s ^①	1.30 s ^①

^①The chemical shifts of methyls in the literature were not assigned, and here, the placements are only to display the data.

Table 15-1-5: ¹H NMR spectroscopic data of marine terpenoids 15-1-10~15-1-12.

H	15-1-10 (CDCl ₃)	15-1-10 (C ₅ D ₅ N)	15-1-11	15-1-12
1	α 1.86 dt(13.2, 4.4) β 1.17 br d(13.2)	α 1.88 m β –	1.56 m 1.39 m	α 1.71 m β 1.34 td(13.2, 3.2)
2	α 1.56 m β 1.61 m	–	1.52 m 1.32 m	α 1.56 m β 2.10 dt(14.0, 4.0)
3	α 1.61 m, β 1.53 m	–	2.43 m, 2.39 m	α 1.95 m, β 1.52 m
4	1.94 m	2.16 m		
6	6.86 s	6.23 br s	5.92 s	6.04 s
8			2.43 m	α 2.39 br dt(17.6, 3.6) β 2.61 ddd(17.6, 14.4, 4.8)
9	α 2.93 d(16.8) β 2.05 d(16.8)	α 2.63 d(16.8) β 2.19 d(16.8)	α 1.78 m β 1.98 m	α 1.74 m β 1.86 dd(14.4, 4.8)
11				1.46 s
12	1.42 s	1.64 s	1.34 s	1.42 s
13	1.43 s	1.72 s	1.35 s	
14	1.05 s	1.11 d(6.4)	1.08 s	
15	1.00 d(6.4)	1.12 d(6.4)	2.13 s	
OMe			3.10 s	

Table 15-1-6: ¹H NMR spectroscopic data of marine terpenoids 15-1-13~15-1-15.

H	15-1-13	15-1-14	15-1-15
1ax	1.40 td(13.8, 4.2)	1.37 td(13.8, 4.8)	1.40 m(ov)
1eq	2.05 br ddd(13.2, 4.8, 2.4)	1.98 ddd(13.8, 3.6, 1.2)	2.05 dt(13.2, 3.0)
2	1.60 m(ov)	ax 1.72 m, eq 1.50 m	ax 1.50 m(ov), eq 1.40 m(ov)
3	ax 1.40 m(ov), eq 1.30 m	ax 1.40 m, eq 1.30 m	ax 1.40 m(ov), eq 1.30 m
4	1.49 m	1.55 m	1.50 m(ov)
6ax	1.85 td(14.4, 4.8)	1.84 td(14.4, 4.8)	1.81 td(14.4, 5.4)
6eq	1.78 ddd(14.4, 6.0, 2.4)	1.78 ddd(14.4, 5.4, 2.4)	1.74 ddd(14.4, 6.6, 1.2)

Table 15-1-6 (continued)

H	15-1-13	15-1-14	15-1-15
7ax	2.70 dddd(15.6, 14.4, 6.0, 3.0)	2.63 dddd(15.0, 14.4, 6.0, 3.0)	2.55 dddd(18.0, 14.4, 6.6, 3.0)
7eq	2.44 dddd(15.6, 4.8, 2.4, 0.6)	2.40 dddd(15.0, 4.8, 2.4, 0.6)	3.61 ddt(18.0, 5.4, 1.2)
11	5.95 dd(3.0, 0.6)	5.75 dd(3.0, 0.6)	6.50 dd(3.0, 1.2)
13	0.82 d(7.2)	0.83 d(7.2)	0.82 d(6.6)
14	0.89 s	0.91 s	0.91 s
15	1.06 s	1.12 s	1.01 s
OMe		3.70 s	

Table 15-1-7: ^1H NMR spectroscopic data of marine terpenoids 15-1-16 and 15-1-17.

H	15-1-16	15-1-17	H	15-1-16	15-1-17
1	1.74 m 1.20 dt(13.2, 3.0)	α 1.15 β 1.78 d(12.5)	11	1.54 s	0.89 s
2	2.04 br s	1.52	12	0.96 s	0.83 s
3	5.15 s	α 1.41, β 1.43	13	0.92 s	1.20 s
5		0.94	14	0.99 d(7.4)	0.88 s
6	1.44 dt(13.2, 3.6) 1.27 dd(13.8, 3.6)	1.53	15	2.28 d(12.6) 2.52 d(12.6)	6.58 d(7.0)
7	1.70 dd(13.8, 3.6) 0.99 d(7.4)	α 2.16 d(13.5) β 1.52	17	7.99 s(OH)	
9	1.47 m	1.96 d(6.4)	18		5.53 s
10	1.89 ddd(12.6, 4.2, 1.8)		19	5.62 s	
			20		4.22 d(4.0)
			21		4.69 d(4.0)

Table 15-1-8: ^1H NMR spectroscopic data of marine terpenoids 15-1-18, 15-1-25, and 15-1-26.

H	15-1-18	15-1-25	15-1-26	H	15-1-18	15-1-25	15-1-26
1	2.74 s	2.73 s	5.28 s	12	5.12 s	4.69 s	
4	3.57 t(9)	3.76 t(8)	3.80 m	13	2.33 s	4.71 s	2.42 s
5	3.15 t(9)	3.30 t(8)	3.54 m, 3.28 m	14	1.14 s	1.16 s	1.45 s
8	7.01 s	7.19 s	7.15 s	15	1.14 s	1.16 s	0.44 s
10	2.69 s	2.82 s	2.48 d(15), 3.30 m	2'	2.08 s		

Table 15-1-9: ^1H NMR spectroscopic data of marine terpenoids **15-1-19**~**15-1-24**.

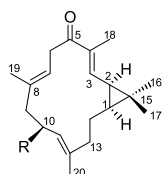
H	15-1-19	15-1-20	15-1-21	15-1-22	15-1-23	15-1-24
1	2.74 s	2.73 s	2.74 s	2.74 s	2.73 s	2.73 s
4	3.57 t(9)	4.15 t(8)	4.15 t(8)	4.15 m	3.63 t(9)	3.62 t(8)
5	3.15 t(9)	3.03 t(8)	3.03 t(8)	3.02 m	3.20 t(9)	3.20 t(8)
8	7.26 s	7.01 s	7.01 s	7.01 s	7.20 s	7.19 s
10	2.69 s	2.69 s	2.69 s	2.69 s	2.77 s	2.77 s
12	5.13 s	5.15 s	5.15 s	5.15 s	5.13 s	5.13 s
13	2.42 s	2.35 s	2.35 s	2.36 s	5.14 s	5.14 s
14	1.14 s	1.13 s	1.14 s	1.14 s	1.15 s	1.14 s
15	1.14 s	1.13 s	1.14 s	1.14 s	1.15 s	1.14 s
2'	2.30 m	2.29 m	2.06 s	2.06 s	2.08 s	2.10 s
3'	1.62 m	1.64 m				
4'	0.94 t(7)	0.93 t(7)				
2''		2.29 m	2.07 s	2.35 m	2.10 s	2.31 t(7)
3''		1.64 m		1.67 m		1.66 (7)
4''		0.94 t(7)		0.94 t(7)		0.94 t(7)

Table 15-1-10: ^1H NMR spectroscopic data of marine terpenoids **15-1-27** and **15-1-28**.

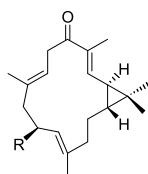
H	15-1-27	15-1-28	H	15-1-27	15-1-28
1	2.06 m, 1.49 m	2.08 m, 1.49 m	1'	2.01 m, 1.59 m	1.99 m, 1.64 m
2	2.04 m, 1.95 m	2.04 m, 1.92 m	2'	1.90 m, 1.2~1.3 m	2.11 m
3	5.15 br s	5.15 br s	3'	2.35 ddd(13.5, 13.5, 4.8)	5.15 br s
6	1.64 m, 1.11 m	1.65 m, 1.11 m		2.11 m	
7	1.38 m, 1.34 m	1.37 m	6'	1.50 m, 1.21 m	1.60 m, 0.94 m
8	1.32 m	1.32 m	7'	1.42 m	1.37 m
10	1.11 d(12.0)	1.11 d(11.6)	8'	1.25 m	1.22 m
11	1.55 br s	1.55 br s	10'	0.91 d(11.8)	1.17 d(11.8)
12	1.02 s	1.02 s	11'	4.46 s, 4.41 s	1.53 s
13	0.99 d(6.3)	1.00 d(5.7)	12'	1.07 s	1.03 s
14	0.85 s	0.86 s	13'	0.99 d(6.3)	1.00 d(6.3)
15	2.61 d(13.9)	2.62 d(13.7)	14'	0.85 s	0.86 s
	2.47 d(13.9)	2.49 d(13.7)	15'	2.59 br s	2.70 d(14.5), 2.61 d(14.5)
19	5.21 s	5.24 s	17'	8.45 (OH)	
NH	5.8~6.4 br s	5.88 br s	19'	11.06 s(OH)	11.07 s(OH)
OMe	3.91 s	3.92 s	21'	7.54 s	7.60 s

Table 15-1-11: Compounds, MFs, and test solvents of marine terpenoids **15-1-29~15-1-50**.

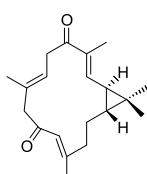
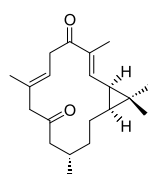
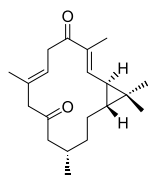
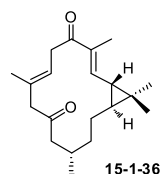
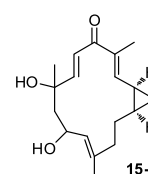
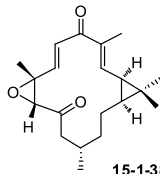
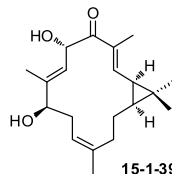
No.	Compounds	MFs	Test solvents	References
15-1-29	depressin	$C_{20}H_{30}O$	$CDCl_3$	[9]
15-1-30	10-hydroxydepressin	$C_{20}H_{30}O_2$	$CDCl_3$	[9]
15-1-31	1- <i>epi</i> -depressin	$C_{20}H_{30}O$	$CDCl_3$	[9]
15-1-32	1- <i>epi</i> -10-hydroxydepressin	$C_{20}H_{30}O_2$	$CDCl_3$	[9]
15-1-33	1- <i>epi</i> -10-oxodepressin	$C_{20}H_{28}O_2$	$CDCl_3$	[9]
15-1-34	10-oxo-11,12-dihydrodepressin	$C_{20}H_{30}O_2$	$CDCl_3$	[9]
15-1-35	1- <i>epi</i> -10-oxo-11,12-dihydrodepressin	$C_{20}H_{30}O_2$	$CDCl_3$	[9]
15-1-36	2- <i>epi</i> -10-oxo-11,12-dihydrodepressin	$C_{20}H_{30}O_2$	$CDCl_3$	[9]
15-1-37	8,10-dihydroxy- <i>iso</i> -depressin	$C_{20}H_{30}O_3$	$CDCl_3$	[9]
15-1-38	microclavatin	$C_{20}H_{28}O_3$	—	[10]
15-1-39	—	$C_{20}H_{30}O_3$	$CDCl_3$	[11]
15-1-40	agrostistachin	$C_{20}H_{30}O_3$	$CDCl_3$	[12]
15-1-41	14-dehydroagrostistachin	$C_{20}H_{28}O_3$	$CDCl_3$	[13]
15-1-42	17-hydroxyagrostistachin	$C_{20}H_{30}O_4$	$CDCl_3$	[13]
15-1-43	agroskerin	$C_{20}H_{30}O_3$	$CDCl_3$	[13]
15-1-44	koumbalone A	$C_{20}H_{28}O_3$	$CDCl_3$	[14]
15-1-45	koumbalone B	$C_{20}H_{28}O_3$	$CDCl_3$	[14]
15-1-46	yuexiandajisu A	$C_{20}H_{30}O_3$	$CDCl_3$	[15]
15-1-47	yuexiandajisu B	$C_{20}H_{30}O_2$	$CDCl_3$	[15]
15-1-48	hookerianolide A	$C_{20}H_{28}O_6$	CD_3OD	[16]
15-1-49	hookerianolide B	$C_{20}H_{28}O_5$	$DMSO-d_6$	[16]
15-1-50	hookerianolide C	$C_{22}H_{32}O_6$	$DMSO-d_6$	[16]

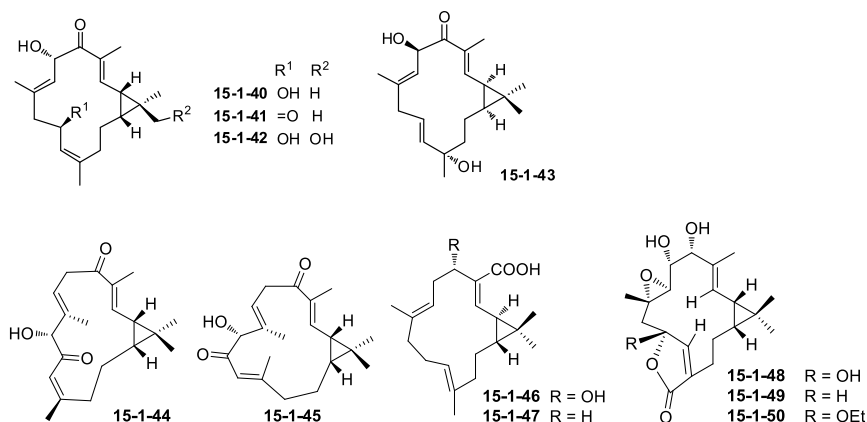


15-1-29 R = H
15-1-30 R = OH



15-1-31 R = H
15-1-32 R = OH

**15-1-33****15-1-34****15-1-35****15-1-36****15-1-37****15-1-38****15-1-39**

**Table 15-1-12:** ^1H NMR spectroscopic data of marine terpenoids 15-1-29~15-1-32.

H	15-1-29	15-1-30	15-1-31	15-1-32
1	1.15 m	1.15 m	0.71 m	0.57 m
2	1.50 dd(10.2, 8.7)	1.50 dd(10.2, 8.4)	1.08 m	1.03 m
3	6.37 d(10.2)	6.30 d(10.5)	6.11 d(10.2)	6.04 d(10.2)
6	3.55 dd(13.8, 4.4)	3.50 dd(13.8, 8.4)	3.71 dd(13.8, 11.1)	3.63 dd(14.3, 11.0)
	2.97 dd(13.8, 5.7)	3.02 dd(13.8, 5.7)	2.83 br d(13.8)	2.89 br d(14.3)
7	5.08 t(6.6)	5.11 t(6.6)	5.21 br d(11.1)	5.27 (ov)
9	2.15 m, 2.00 m	2.39 m, 2.07 m	2.18 m, 2.10 m	2.48 br d(11.4), 2.06 m
10	2.17 m, 1.96 m	4.38 td(9.3, 9.3)	2.28 m, 2.07 m	4.31 td(9.6, 3.8)
11	4.84 t(5.4)	5.06 d(9.3)	4.88 br d(8.4)	5.26 (ov)
13	2.20 m, 1.75 m	2.38 m, 1.80 m	2.17 m, 1.93 m	2.33 m, 2.13 m
14	2.05 m, 0.80 m	2.07 m, 1.19 m	1.88 m, 1.03 m	1.99 m, 1.27 m
16	1.16 s	1.18 s	1.16 s	1.15 s
17	1.09 s	1.06 s	1.11 s	1.11 s
18	1.87 s	1.83 s	1.80 s	1.77 s
19	1.56 s	1.66 s	1.59 s	1.71 s
20	1.56 s	1.63 s	1.59 s	1.59 s

Table 15-1-13: ^1H NMR spectroscopic data of marine terpenoids 15-1-33~15-1-36.

H	15-1-33	15-1-34	15-1-35	15-1-36
1	0.65 m	0.97 m	0.75 m	0.65 m
2	1.10 m	1.42 m	1.15 m	1.14 m
3	6.12 d(9.9)	6.35 d(10.8)	6.05 d(10.5)	6.08 d(10.5)
6	3.80 dd(13.5, 10.8)	3.85 dd(13.5, 10.8)	3.72 dd(12.0, 14.7)	3.63 dd(14.7, 10.5)
	2.91 d(13.5)	2.90 br d(13.5)	2.95 (ov)	3.05 dd(14.7, 3.0)
7	5.35 br d(10.8)	5.42 dd(10.8, 1.2)	5.42 dd(12.0, 1.8)	5.42 dd(10.5, 3.0)

Table 15-1-13 (continued)

H	15-1-33	15-1-34	15-1-35	15-1-36
9	3.10 d(13.2) 2.91 d(13.2)	3.07 d(11.1) 2.80 d(11.1)	3.03 (ov) 2.93 (ov)	3.03 s 3.03 s
11	6.03 s	2.60 dd(18.0, 9.0) 2.12 d(18.0)	2.70 d(17.1) 2.05 d(17.1)	2.35 m
12		2.08 m	2.02 m	2.10 m
13	2.35 m, 1.98 m	1.40 m, 0.80 m	1.45 m, 1.25 m	1.65 m, 1.30 m
14	2.02 m, 1.18 m	1.50 m, 0.60 m	1.83 m, 1.20 m	2.00 m, 1.07 m
16	1.17 s	1.16 s	1.12 s	1.13 s
17	1.11 s	1.15 s	1.13 s	1.15 s
18	1.84 s	1.83 s	1.82 s	1.83 s
19	1.61 s	1.79 s	1.61 s	1.64 s
20	2.17 s	0.95 d(7.2)	0.90 d(6.6)	0.88 d(6.6)

Table 15-1-14: ¹H NMR spectroscopic data of marine terpenoids 15-1-37~15-1-40.

H	15-1-37	15-1-38	15-1-39	15-1-40
1	1.24 m	1.05 m	1.16~1.25 m	1.11 m
2	1.52 m	1.45 dd(10.5, 8.0)	1.53 dd(8.1, 9.3)	1.51 dd(10.0, 8.0)
3	6.39 d(10.5)	6.00 d(10.5)	6.28 d(9.3)	6.25 d(10.0)
6	6.55 d(16.2)	6.44 d(16.5)	5.25 d(9.6)	5.22 d(10.0)
7	6.10 d(16.2)	5.81 d(16.5)	5.17 d(9.6)	5.11 br d
9	2.40 dd(15.0, 3.6) 1.91 m	3.67 s	4.11 dd(9.0, 6.6)	2.44 dd(12.0, 2.5) 2.01 m
10	4.85 m		2.29 m	4.18 m
11	5.67 d(10.2)	2.70 dd(2.5, 15.0) 2.38 dd(10.5, 15.0)	4.68 dd(6.3)	5.11 br d
12		1.85 m		
13	2.20 m, 1.78 m	1.26 m, 1.16 m	2.03~2.44 m, 1.60~1.80 m	2.25 m, 1.78 m
14	1.91 m, 1.40 m	1.82 m, 0.90 m	1.16~1.25 m, 0.70~0.85 m	2.01 m, 0.83 m
16	1.27 s	1.14 s	1.01 s	0.96 s
17	1.15 s	0.94 s	1.16 s	1.14 s
18	1.87 s	1.89 s	1.90 s	1.87 d(0.7)
19	1.45 s	1.63 s	1.70 br s	1.76 d(1.5)
20	1.69 s	1.08 d(7.5)	1.59 br s	1.70 br s

Table 15-1-15: ¹H NMR spectroscopic data of marine terpenoids 15-1-41~15-1-44.

H	15-1-41	15-1-42	15-1-43	15-1-44
1	1.15 m	1.31 m	1.12 m	1.10 m
2	1.57 dd(11.0, 8.0)	1.73 dd(11.0, 8.0)	1.52 dd(11.0, 9.0)	1.53 dd(11.2, 8.5)
3	6.19 d(11.0)	6.32 d(11.0)	6.28 d(11.0)	6.07 dd(11.2, 1.5)

Table 15-1-15 (continued)

H	15-1-41	15-1-42	15-1-43	15-1-44
6	5.21 m	5.29 d(9.0)	5.19 d(10.0)	3.82 dd(14.0, 11.5) 3.00 br d(14.0)
7	5.28 d(10.0)	5.19 m	5.08 d(10.0)	5.51 br d(11.5)
9	3.18 d(10.0)	2.53 dd(12.0, 2.9)	2.71 dd(13.0, 7.0)	4.60 d(3.0)
	2.85 d(10.0)	2.10 m	2.59 dd(13.0, 7.0)	
10		4.27 m	5.70 m	
11	5.88 s	5.19 m	5.40 d(16.0)	5.78 s
13	3.42 m	2.35 m	1.60 m	3.86 m
	1.94 m	1.87 m	1.37 m	1.76 dt(2.2, 12.0)
14	0.91 m, 2.11 m	0.95 m, 2.10 m	0.90 m, 1.43 m	2.15 br d(14.0) 0.92 br q(12.0)
16	0.99 s	1.09 s	1.07 s	1.03 s
17	1.18 s	3.48 s	1.15 s	1.18 s
18	1.93 d(1.1)	1.94 s	1.92 s	1.84 d(1.5)
19	1.91 d(1.1)	1.84 d(2.7)	1.87 br s	1.60 t(3.0)
20	1.88 d(1.2)	1.78 br s	1.29 s	1.90 d(1.5)
OH	4.30 d(6-OH)			

Table 15-1-16: ¹H NMR spectroscopic data of marine terpenoids 15-1-45~15-1-48.

H	15-1-45	15-1-46	15-1-47	15-1-48
1	1.19 ddd(10.0, 8.5, 2.0)	0.75 m	0.67 m	0.76 t(8.7)
2	1.60 dd(10.8, 8.5)	2.06 m	2.10~2.13 m	1.53 dd(11.7, 8.7)
3	6.26 dq(10.8, 1.8)	5.74 d(11.1)	5.57 d(10.8)	5.55 d(11.7)
5		4.15 dd(5.3, 11.0)	2.72 m, 2.10~2.13 m	4.07 d(8.8)
6	3.80 dd(10.0, 14.0)	2.48 m	2.36 m	3.57 t(8.8)
	2.97 br d(14.0, 1.6)	2.67 m		
7	5.57 br d(10.0)	4.88 dd(5.2, 7.2)	4.29 br t	2.64 d(8.8)
9	4.50 s	2.04 m	2.10~2.13 m	α 2.78 d(14.1) β 1.24 d(14.1)
10		2.12 m	2.10~2.13 m	
11	6.11 q(1.7)	5.02 br t	5.01 br t	7.12 t(2.0)
13	2.32 dt(11.0, 8.0)	2.14 m	2.10~2.13 m	α 2.22~2.32 m β 2.49~2.55 m
	1.90 dd(10.0, 11.0)			
14	2.23 m	1.19 m	1.01 m	α 2.00 dt(14.7, 3.8)
	0.79 br q(12.0)	1.95 m	1.92 m	β 1.44~1.50 m
16	1.06 s	1.15 s	1.12 s	1.08 s
17	1.16 s	1.19 s	1.12 s	1.14 s
18	1.92 d(1.8)			1.78 s
19	1.40 t(1.6)	1.58 s	1.58 s ^①	1.53 s
20	2.13 d(1.7)	1.57 s	1.54 s ^①	

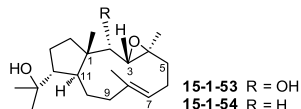
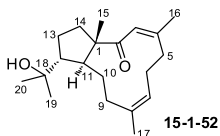
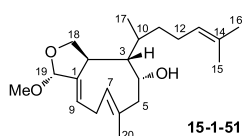
^①The chemical shifts were not assigned in the literature, and here, the placements are only to display the data.

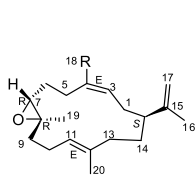
Table 15-1-17: ^1H NMR spectroscopic data of marine terpenoids **15-1-49** and **15-1-50**.

H	15-1-49	15-1-50	H	15-1-49	15-1-50
1	0.73 t(8.7)	0.78 t(8.8)	13 α	2.15~2.27 m	2.26~2.36 m
2	1.53 dd(11.0, 8.8)	1.55 dd(11.2, 8.8)	13 β	2.42~2.48 m	2.45~2.51 m
3	5.59 d(11.0)	5.60 d(11.2)	14 α	1.97 dt(14.7, 3.9)	1.92 dt(14.6, 3.8)
5	4.06 d(8.9)	4.07 d(8.9)	14 β	1.50~1.56 m	1.50~1.60 m
6	3.51 t(8.9)	3.54 t(8.9)	16	1.02 s	1.04 s
7	2.61 d(8.9)	2.63 d(8.9)	17	1.12 s	1.12 s
9 α	2.67 d(12.5, 4.7)	2.74 d(13.8)	18	1.76 s	1.76 s
9 β	0.88 t(12.5)	1.24 d(13.8)	19	1.46 s	1.48 s
10	5.05 d(12.5)		OEt		3.41 q(7.0), 1.15 t(7.0)
11	7.52 d(1.3)	7.12 t(2.0)			

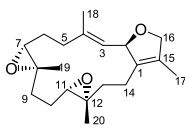
Table 15-1-18: Compounds, MFs, and test solvents of marine terpenoids **15-1-51**~**15-1-68**.

No.	Compounds	MFs	Test solvents	References
15-1-51	—	$\text{C}_{21}\text{H}_{34}\text{O}_3$	CDCl_3	[17]
15-1-52	(1 S^* ,3 Z ,7 Z ,11 S^* ,12 R^*)-18-hydroxydolabella-3,7-dien-2-one	$\text{C}_{20}\text{H}_{32}\text{O}_2$	CDCl_3	[17]
15-1-53	—	$\text{C}_{20}\text{H}_{34}\text{O}_3$	CDCl_3	[17]
15-1-54	—	$\text{C}_{20}\text{H}_{34}\text{O}_2$	CDCl_3	[17]
15-1-55	knightol	$\text{C}_{20}\text{H}_{32}\text{O}_2$	CDCl_3	[18]
15-1-56	knightol acetate	$\text{C}_{22}\text{H}_{34}\text{O}_3$	CDCl_3	[18]
15-1-57	knightal	$\text{C}_{20}\text{H}_{30}\text{O}_2$	CDCl_3	[18]
15-1-58	11,12-epoxy-sarcophytoxide	$\text{C}_{20}\text{H}_{30}\text{O}_3$	CDCl_3	[19]
15-1-59	durumolide F	$\text{C}_{22}\text{H}_{32}\text{O}_6$	CDCl_3	[20]
15-1-60	durumolide G	$\text{C}_{24}\text{H}_{34}\text{O}_8$	CDCl_3	[20]
15-1-61	durumolide H	$\text{C}_{26}\text{H}_{36}\text{O}_9$	CDCl_3	[20]
15-1-62	durumolide I	$\text{C}_{24}\text{H}_{32}\text{O}_8$	CDCl_3	[20]
15-1-63	durumolide J	$\text{C}_{20}\text{H}_{28}\text{O}_4$	CDCl_3	[20]
15-1-64	durumolide K	$\text{C}_{20}\text{H}_{26}\text{O}_5$	CDCl_3	[20]
15-1-65	durumolide L	$\text{C}_{20}\text{H}_{24}\text{O}_5$	CDCl_3	[20]
15-1-66	durumhemiketalolide A	$\text{C}_{20}\text{H}_{28}\text{O}_5$	CDCl_3	[21]
15-1-67	durumhemiketalolide B	$\text{C}_{22}\text{H}_{30}\text{O}_6$	CDCl_3	[21]
15-1-68	durumhemiketalolide C	$\text{C}_{22}\text{H}_{30}\text{O}_6$	CDCl_3	[21]

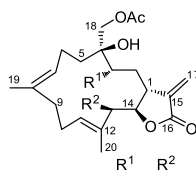




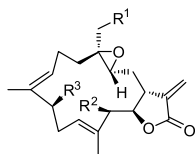
15-1-55 R = CH₂OH
15-1-56 R = CH₂OAc
15-1-57 R = CHO



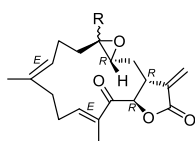
15-1-58



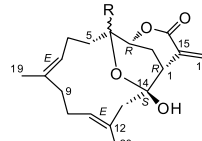
15-1-59 OH H
15-1-60 OAc OH
15-1-61 OAc OAc



15-1-62 OAc OAc OH
15-1-63 H OH H



15-1-64 R = (S)-CH₂OH
15-1-65 R = (R)-CHO



15-1-66 R = (S)-CH₂OH
15-1-67 R = (S)-CH₂OAc
15-1-68 R = (R)-CH₂OAc

Table 15-1-19: ¹H NMR spectroscopic data of marine terpenoids **15-1-51~15-1-54**.

H	15-1-51	15-1-52	15-1-53	15-1-54
2	3.02 br d(6.0)		4.08 d(2.0)	1.53 m
3	1.99 m	6.36 s	2.86 d(2.0)	2.93 dd(10.0, 3.0)
4	4.26 m			
5	2.14 dd(13.5, 4.0)	1.96 m	1.18 m	1.18 m
	2.31 dd(13.5, 2.0)	3.12 br d(11.0)	2.05 m	2.11 m
6		1.80 m	2.11 m	2.10 m
		2.72 dd(12.5, 6.0)	2.38 m	2.33 m
7	5.34 dd(11.5, 4.0)	4.97 br d(12.5)	5.07 d(12.0)	5.08 br d(10.0)
8	2.74 ddd(16.0, 7.5, 4.0)			
	3.14 ddq(16.0, 11.5, 1.5)			
9	5.84 dddd	1.62 m	2.06 m, 2.41 m	2.07 m, 2.32 m
	(7.5, 1.5, 1.5, 1.5)	2.21 td(14.5, 2.0)		
10	2.01 m	1.40 m, 2.03 m	1.73 m	1.57 m, 1.73 m
11	1.09 m, 1.20 m	2.27 ddd(12.0, 8.0, 3.0)	1.97 m	1.54 m
12	1.92 dt(7.5, 7.5)	1.76 m	1.72 m	1.65 m
13	5.08 hept(7.5, 1.5)	1.62 m, 1.95 m	1.61 m, 1.97 m	1.36 m, 1.55 m
14		1.41 m, 2.00 m	1.63 m, 1.90 m	1.41 m, 1.52 m
15	1.59 br s	1.07 s	1.26 s	1.26 s
16	1.68 br s	1.81 s	1.52 s	1.27 s
17	1.09 d(7.0)	1.65 s	1.74 s	1.69 s
18	3.81 dd(9.5, 6.0), 4.09 d(9.5)			
19	5.19 br d(1.5)	1.26 s	1.26 s	1.17 s
20	1.86 br s	1.27 s	1.12 s	1.24 s
OMe	3.40 s			

Table 15-1-20: ¹H NMR spectroscopic data of marine terpenoids **15-1-55~15-1-59**.

H	15-1-55	15-1-56	15-1-57	15-1-58	15-1-59
1	1.97 m	1.96 m	2.09 m		2.97 m
2	2.37 m, 1.82 m	2.38 m, 1.82 m	2.70 m, 2.10 m	5.44 m	1.93 m, 1.64 m
3	5.51 brt(7.8)	5.56 t(7.9)	6.49 dd(9.8, 5.0)	5.20 d(10.1)	3.49 brt(8.3)
5	2.22 m, 2.33 m	2.23 m, 2.30 m	2.45 dt(14.3, 7.4) 2.33 dt(13.7, 6.9)	α 2.37 m β 2.82 m	1.68 m, 1.86 m
6	2.02 m, 1.49 m	1.88 m, 1.46 m	1.96 m, 1.47 m	α 1.69 m, β 1.87 m	2.20 m
7	2.76 dd(7.5, 3.4)	2.74 dd(7.4, 3.4)	2.65 dd(7.3, 4.1)	2.78 dd(4.2, 4.2)	5.26 brt(6.9)
9	2.04 m, 1.42 m	2.00 m, 1.42 m	1.88 m, 1.46 m	α 1.18 m, β 2.20 m	2.18 m
10	2.16 m, 1.59 m	2.16 m, 1.59 m	2.13 m	α 1.36 m, β 2.02 m	2.39 m, 2.17 m
11	5.19 brt(6.6)	5.16 t(6.7)	5.21 brt(6.6)	2.52 dd(2.8, 10.4)	5.09 br d(4.9)
13	2.22 m 2.17 m	2.22 m 2.14 m	2.14 m 2.07 m	α 0.95 m β 2.24 m	2.57 br d(12.4) 2.16 m
14	1.59 m, 1.45 m	1.58 m, 1.44 m	1.64 m, 1.53 m	2.46 m	4.32 dt(9.9, 2.8)
16	1.68 s	1.66 s	1.69 s	4.50 br s	
17	4.73 br s 4.61 br s	4.71 br s 4.60 br s	4.78 br s 4.64 br s	1.67 s	6.29 d(1.3) 5.68 d(1.9)
18	4.09 d(12.6) 4.03 d(12.6)	4.54 d(12.3) 4.43 d(12.3) 2.06 s(OAc)	9.33 s	1.75 s	4.20 d(11.8) 4.13 d(11.8) 2.11 s(OAc)
19	1.21 s	1.20 s	1.14 s	1.32 s	1.64 s
20	1.61 s	1.59 s	1.59 s	1.26 s	1.76 s

Table 15-1-21: ¹H NMR spectroscopic data of marine terpenoids **15-1-60~15-1-64**.

H	15-1-60	15-1-61	15-1-62	15-1-63	15-1-64
1	2.63 m	2.75 m	2.82 m	2.77 m	3.06 m
2	2.00 m, 2.07 m	1.96 m	1.78 dt(14.4, 2.9) 1.56 m	1.87 dt(14.6, 2.4) 1.40 m	1.78 m, 1.95 m
3	5.22 dd(8.0, 5.5) 2.06 d(OAc)	5.17 dd(7.4, 5.1) 2.07 s(OAc)	2.82 m	2.70 dd(7.3, 2.3)	3.04 m
5	1.64 m, 1.76 m	1.70 m, 1.83 m	1.23 td(13.0, 2.6) 2.46 m	1.24 m, 2.08 m	1.65 m, 2.13 m
6	2.25 m, 2.20 m	2.20 m	2.35 m, 2.19 m	2.23 m, 2.10 m	2.18 m, 2.10 m
7	5.08 brt(6.5)	5.06 brt(6.8)	5.37 br dd(9.6, 3.9)	5.03 br d(4.3)	5.18 brt(5.6)
9	2.20 m	2.20 m	4.11 dd(10.4, 2.7)	2.35 m, 2.12 m	2.27 m
10	2.46 m, 2.25 m	2.40 m, 2.25 m	2.67 m, 2.41 m	2.58 m, 2.52 m	2.58 m, 2.44 m
11	5.44 dd(7.5, 3.0)	5.54 br d(6.6)	5.52 br dd(8.3, 5.6)	5.47 br dd(9.7, 3.1)	6.68 t(6.8)

Table 15-1-21 (continued)

H	15-1-60	15-1-61	15-1-62	15-1-63	15-1-64
13	3.96 d(8.5)	5.10 d(8.5) 2.08 s(OAc)	5.28 d(8.5) 2.12 s(OAc)	4.05 br s	
14	4.24 dd(8.5, 6.0)	4.33 dd(8.5, 4.8)	4.17 d(8.5)	4.06 d(6.0)	5.57 d(2.0)
17	6.29 d(2.5) 5.85 d(2.5)	6.29 d(1.9) 5.78 d(1.3)	6.33 d(2.9) 6.03 d(2.5)	6.32 d(3.2) 6.08 d(2.8)	6.28 d(2.0) 5.62 d(1.6)
18	4.14 d(12.0) 4.05 d(12.0) 2.12 s(OAc)	4.13 d(11.8) 4.05 d(11.8) 2.12 s(OAc)	4.41 d(12.2) 3.85 d(12.2) 2.15 s(OAc)	1.24 s	3.72 d(11.6) 3.67 d(11.6)
19	1.67 s	1.67 s	1.71 s	1.64 s	1.62 s
20	1.74 s	1.74 s	1.72 s	1.73 s	1.84 s

Table 15-1-22: ¹H NMR spectroscopic data of marine terpenoids 15-1-65~15-1-68.

H	15-1-65	15-1-66	15-1-67	15-1-68
1	3.12 dt(12.4, 2.4)	3.46 t(9.0)	3.43 t(9.0)	3.37 dd(9.5, 2.0)
2	2.12 m 1.77 m	2.36 m 2.03 m	2.38 ddd(14.0, 9.0, 6.5) 2.00 ddd(14.0, 10.0, 9.0)	2.30 m 1.89 ddd(13.0, 5.5, 2.0)
3	3.35 dd(8.6, 2.4)	4.70 dd(10.5, 6.0)	4.48 dd(10.0, 6.5)	4.16 dd(11.0, 5.5)
5	2.33 m 2.01 ddd(12.8, 6.4, 4.0)	1.74 dd(9.5, 4.0) 1.64 m	1.75 m 1.66 m	2.27 m 1.71 m
6	2.17 m, 2.15 m	2.14 m	2.23 m, 2.08 m	2.14 m, 2.08 m
7	5.15 br t(6.0)	5.09 t(7.5)	5.14 t(7.0)	5.09 t(6.0)
9	2.32 m, 2.27 m	2.18 m, 2.10 m	2.17 m, 2.11 m	2.16 m, 2.10 m
10	2.64 m, 2.42 m	2.35 m, 2.16 m	2.27 m, 2.18 m	2.35 m, 2.22 m
11	6.63 dd(6.8, 4.8)	5.35 t(7.5)	5.32 t(6.5)	5.10 br t(6.0)
13		2.77 d(13.5) 2.30 d(13.5)	2.79 d(14.0) 2.23 d(14.0)	2.61 d(13.5) 2.38 d(13.5)
14	5.51 d(2.0)			
17	6.34 d(2.0) 5.67 d(1.6)	6.32 d(1.5) 5.72 d(1.5)	6.30 d(2.0) 5.70 d(2.0)	6.35 d(2.0) 5.72 d(2.0)
18	9.42 s	3.76 d(11.0) 3.47 d(11.0)	4.34 d(12.0) 4.00 d(12.0) 2.09 s(OAc)	4.15 d(11.5) 4.09 d(11.5) 2.13 s(OAc)
19	1.61 s	1.66 s	1.63 s	1.52 s
20	1.86 s	1.75 s	1.73 s	1.77 s

Table 15-1-23: Compounds, MFs, and test solvents of marine terpenoids 15-1-69~15-1-89.

No.	Compounds	MFs	Test solvents	References
15-1-69	simplexin A	C ₂₆ H ₄₂ O ₆	CDCl ₃	[22]
15-1-70	simplexin B	C ₂₆ H ₄₄ O ₇	CDCl ₃	[22]
15-1-71	simplexin H	C ₂₈ H ₄₆ O ₉	CDCl ₃	[22]

Table 15-1-23 (continued)

No.	Compounds	MFs	Test solvents	References
15-1-72	simplexin I	$C_{26}H_{42}O_9$	$CDCl_3$	[22]
15-1-73	simplexin C	$C_{32}H_{52}O_{11}$	$CDCl_3$	[22]
15-1-74	simplexin D	$C_{34}H_{56}O_{11}$	$CDCl_3$	[22]
15-1-75	simplexin E	$C_{33}H_{52}O_{11}$	$CDCl_3$	[22]
15-1-76	simplexin F	$C_{28}H_{46}O_{10}$	$CDCl_3$	[22]
15-1-77	simplexin G	$C_{28}H_{46}O_{10}$	$CDCl_3$	[22]
15-1-78	asterolaurin A	$C_{26}H_{36}O_9$	$CDCl_3$	[23]
15-1-79	asterolaurin B	$C_{26}H_{36}O_9$	$CDCl_3$	[23]
15-1-80	asterolaurin C	$C_{26}H_{36}O_9$	$CDCl_3$	[23]
15-1-81	asterolaurin D	$C_{22}H_{32}O_5$	$CDCl_3$	[23]
15-1-82	asterolaurin E	$C_{24}H_{32}O_7$	$CDCl_3$	[23]
15-1-83	asterolaurin F	$C_{22}H_{30}O_5$	$CDCl_3$	[23]
15-1-84	gibberosin O	$C_{21}H_{32}O_4$	$CDCl_3$	[24]
15-1-85	gibberosin P	$C_{19}H_{30}O_3$	$CDCl_3$	[24]
15-1-86	gibberosin Q	$C_{22}H_{34}O_4$	$CDCl_3$	[24]
15-1-87	gibberosin R	$C_{20}H_{32}O_2$	$CDCl_3$	[24]
15-1-88	gibberosin S	$C_{24}H_{36}O_5$	$CDCl_3$	[24]
15-1-89	sinugibberoside F	$C_{20}H_{30}O_4$	$CDCl_3$	[24]

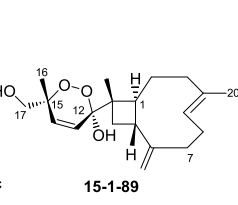
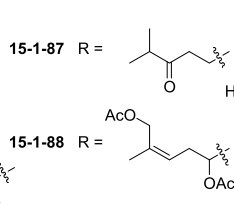
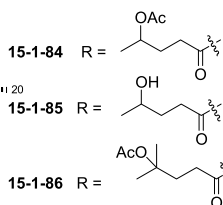
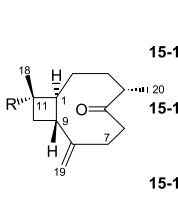
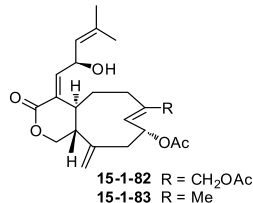
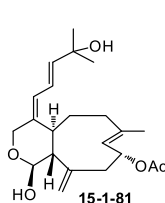
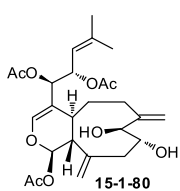
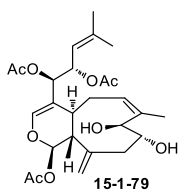
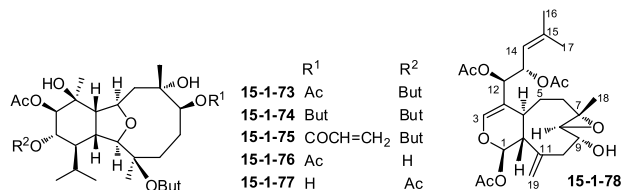
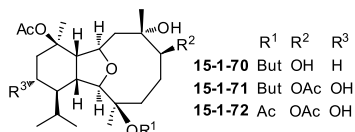
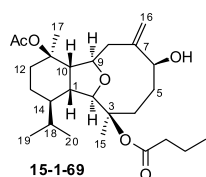


Table 15-1-24: ¹H NMR spectroscopic data of marine terpenoids **15-1-69~15-1-72**.

H	15-1-69	15-1-70	15-1-71	15-1-72
1	2.25 dd(12.0, 7.1)	2.17 dd(11.2, 7.5)	2.13 dd(10.8, 7.0)	2.11 m
2	3.57 s	3.49 s	3.55 s	3.54 s
3				2.10 s(OAc)
4 α	2.14 d(8.5)	1.79 d(14.5)	1.99 m	1.99 m
4 β	1.87 m	2.65 dd(14.5, 8.5)	2.64 dd(14.5, 8.0)	2.62 dd(14.5, 8.5)
5 α	2.10 m	1.57 m	1.54 m	1.51 m
5 β	1.74 m	1.46 m	1.45 m	1.44 m
6	4.30 dd(12.5, 3.0)	4.57 d(6.0)	5.60 d(5.5), 2.08 s(OAc)	5.60 d(5.5), 2.09 s(OAc)
8 α	2.82 dd(13.5, 4.5)	1.86 dd(14.5, 3.5)	1.86 dd(15.0, 3.5)	1.87 dd(15.0, 4.0)
8 β	2.48 d(13.5)	1.99 t(13.0)	2.00 m	2.00 m
9	4.10 dd(10.5, 4.5)	4.06 ddd(11.0, 7.5, 3.5)	4.12 ddd(11.0, 7.5, 3.5)	4.11 ddd(11.0, 8.0, 3.5)
10	3.06 brt(8.8)	3.08 t(7.5)	3.17 t(7.5)	3.26 t(7.5)
11	2.01 s(OAc)	1.99 s(OAc)	1.98 s(OAc)	2.00 s(OAc)
12 α	1.44 m	1.44 m	1.48 m	1.46 m
12 β	2.27 d(12.0)	2.21 d(14.0)	2.32 m	2.26 dd(13.0, 2.5)
13	α 1.46 m β 1.35 t(12.0)	α 1.42 m β 1.33 m	3.88 m	3.90 td(11.0, 3.5)
14	1.20 brt(12.0)	1.14 m	1.22 m	1.19 m
15	1.54 s	1.39 s	1.39 s	1.41 s
16	5.21 s, 5.47 s	1.16 s	1.19 s	1.20 s
17	1.55 s	1.49 s	1.51 s	1.50 s
18	1.86 m	1.73 m	1.71 m	1.72 m
19	0.95 d(7.0)	0.95 d(7.0)	1.18 d(7.0)	1.18 d(6.5)
20	0.78 d(7.0)	0.82 d(7.0)	1.00 d(7.0)	1.00 d(6.5)
3- <i>n</i> -butyrate	0.94 t(7.5) 1.60 tq(7.5, 7.5) 2.16 t(7.5)	1.00 t(7.5) 1.68 tq(7.5, 7.5) 2.30 m, 2.35 m	0.99 t(7.5) 1.67 m 2.30 m, 2.34 m	

Table 15-1-25: ¹H NMR spectroscopic data of marine terpenoids **15-1-73~15-1-75**.

H	15-1-73	15-1-74	15-1-75
1	2.38 m	2.41 dd(11.5, 7.5)	2.42 dd(11.0, 7.5)
2	3.53 s	3.55 s	3.56 s
4 α	1.99 dd(13.5, 11.0)	2.02 dd(14.5, 10.0)	2.05 m
4 β	2.62 dd(13.5, 8.5)	2.65 dd(14.5, 8.5)	2.67 m
5 α	1.50 dd(16.5, 8.5)	1.52 dd(16.7, 8.5)	1.57 m
5 β	1.40 m	1.41 m	1.47 dd(10.5, 5.5)
6	5.58 d(6.0), 2.07 s(OAc)	5.60 d(5.5)	5.69 d(5.5)
8 α	1.80 dd(14.5, 3.5)	1.82 dd(14.5, 3.5)	1.84 dd(14.5, 4.0)

Table 15-1-25 (continued)

H	15-1-73	15-1-74	15-1-75
8 β	1.93 dd(14.5, 11.0)	1.95 dd(14.5, 11.0)	1.97 dd(14.5, 11.0)
9	4.27 ddd(11.0, 7.5, 3.5)	4.29 ddd(11.5, 7.5, 3.5)	4.31 ddd(11.0, 7.5, 3.5)
10	2.60 t(7.5)	2.63 t(7.5)	2.64 t(7.5)
12	5.00 d(10.0), 2.05 s(OAc)	5.03 d(9.5), 2.07 s(OAc)	5.03 d(9.5), 2.07 s(OAc)
13	5.49 t(10.0)	5.50 dd(11.5, 9.5)	5.50 dd(11.0, 9.5)
14	1.73 t(11.5)	1.75 m	1.76 t(11.0)
15	1.37 s	1.39 s	1.40 s
16	1.17 s	1.19 s	1.22 s
17	1.10 s	1.17 s	1.12 s
18	1.69 m	1.71 m	1.74 m
19	0.97 d(7.0)	0.99 d(7.0)	0.99 d(7.0)
20	0.94 d(7.0)	0.96 d(7.0)	0.96 d(7.0)
3- <i>n</i> -butyrate	0.97 t(7.5)	0.97 t(7.5)	0.99 t(7.5)
	1.67 m, 2.25 m, 2.37 m	1.68 m, 2.27 m, 2.39 m	1.68 m, 2.28 m, 2.40 m
6- <i>n</i> -butyrate		0.94 t(7.5), 1.66 m, 2.32 t(7.5)	
6-acrylate			5.84 dd(10.5, 1.5) 6.15 dd(17.5, 10.5) 6.40 dd(17.5, 1.5)
13- <i>n</i> -butyrate	0.92 t(7.5), 1.60 tq(7.5, 7.5) 2.19 td(7.5, 2.0)	0.96 t(7.5), 1.61 tq(7.5, 7.5) 2.21 td(7.5, 2.0)	0.95 t(7.5), 1.60 tq(7.5, 7.5) 2.21 td(7.5, 2.0)

Table 15-1-26: ¹H NMR spectroscopic data of marine terpenoids **15-1-76** and **15-1-77**.

H	15-1-76	15-1-77	H	15-1-76	15-1-77
1	2.33 dd(11.5, 7.0)	2.38 m	13	2.99 dd(11.5, 9.5)	5.47 t(10.0)
2	3.54 s	3.55 s			2.08 s(OAc)
4 α	2.00 dd(15.0, 10.0)	1.82 m	14	1.44 t(11.5)	1.71 m
4 β	2.64 dd(15.0, 7.5)	2.65 dd(13.8, 9.0)	15	1.40 s	1.40 s
5 α	1.52 dd(16.5, 8.5)	1.59 m	16	1.18 s	1.16 s
5 β	1.43 m	1.42 m	17	1.15 s	1.12 s
6	5.60 d(6.0), 2.08 s(OAc)	4.56 d(6.0)	18	1.74 m	1.69 m
8 α	1.83 dd(14.5, 4.0)	1.86 m	19	1.16 d(7.0)	0.99 d(7.0)
8 β	1.94 dd(14.5, 11.0)	1.95 m	20	1.03 d(7.0)	0.96 d(7.0)
9	4.26 ddd(11.0, 7.5, 4.0)	4.24 m	3- <i>n</i> -butyrate	0.99 t(7.5), 1.69 m, 2.28 m, 2.37 m	0.98 t(7.0), 1.67 m, 2.27 m, 2.39 m
10	2.59 t(7.5)	2.60 t(7.0)			
12	4.87 d(9.5) 2.18 s(OAc)	5.01 d(10.0) 2.18 s(OAc)			

Table 15-1-27: ^1H NMR spectroscopic data of marine terpenoids **15-1-78**~**15-1-81**.

H	15-1-78	15-1-79	15-1-80	15-1-81
1	5.88 d(2.1)	5.08 d(9.3)	6.10 d(3.6)	4.60 d(8.2)
3	6.48 s	6.44 d(1.5)	6.44 s	4.31 d(13.7), 4.69 d(13.7)
4a	2.38 m	2.87 m	2.48 m	2.88 d(14.0)
5	α 2.10 m β 1.55 m	2.73 m 2.51 dd(14.1, 5.1)	1.75 m, 2.28 m	α 2.05 m β 1.50 m
6	α 1.25 m, β 2.18 m	5.27 dd(5.1, 12.0)	2.31 m	2.20 m
8	2.97 d(8.4)	4.66 d(8.4)	3.95 br s	2.96 d(8.4)
9	3.74 t(6.6)	4.03 t(7.0)	3.95 m	5.68 t(7.5)
10	α 2.40 m β 2.59 dd(6.7, 8.7)	2.68 m 2.10 m	2.35 m 2.58 m	α 2.35 m β 2.61 dd(7.0, 8.7)
11a	2.28 br s	2.25 d(11.1)	2.55 m	1.71 m
12	5.26 d(6.3)	5.33 d(5.7)	5.27 d(7.2)	5.84 d(14.4)
13	5.65 dd(6.3, 9.3)	5.79 dd(5.7, 9.3)	5.56 dd(6.3, 9.3)	6.44 dd(10.7, 15.1)
14	5.10 m	5.15 d(9.3)	5.10 d(9.0)	5.84 d(14.4)
16	1.70 s	1.76 s	1.72 s	1.36 s
17	1.70 s	1.76 s	1.74 s	1.36 s
18	1.37 s	1.76 s	5.18 s, 5.38 s	1.80 s
19	4.93 s, 5.11 s	4.79 s, 5.48 s	4.97 s, 5.48 s	4.73 s, 4.92 s
OAc	1.97 s, 1.98 s, 2.03 s	2.04 s, 2.05 s, 2.06 s	1.93 s, 1.99 s, 2.03 s	2.10 s

Table 15-1-28: ^1H NMR spectroscopic data of marine terpenoids **15-1-82** and **15-1-83**.

H	15-1-82	15-1-83	H	15-1-82	15-1-83
1 α	3.64 t(12.0)	3.64 t(12.0)	12	6.41 d(7.8)	6.39 d(8.0)
1 β	4.10 dd(12.0, 6.0)	4.10 d(12.0, 5.5)	13	5.22 t(7.8)	5.26 d(8.0)
4a	3.02 d(10.0)	3.0 dd(4.0, 6.5)	14	5.31 d(7.8)	5.31 d(8.0)
5	1.60 m	1.46 m, 1.58 m	16	1.79 s	1.76 s
6	α 2.10 m, β 2.40 m	2.24 m	17	1.75 s	1.79 s
8	5.46 d(8.1)	5.36 d(7.5)	18	4.64 s	1.76 s
9	5.77 t(7.2)	5.67 t(7.0)	19	5.10 s, 5.14 s	5.04 s, 5.08 s
10	α 2.52 d(6.9), β 2.40 m	2.47 d(5.5), 2.41 m	OAc	2.08 s, 2.09 s	2.10 s
11a	2.10 m	2.03 m			

Table 15-1-29: ^1H NMR spectroscopic data of marine terpenoids **15-1-84**~**15-1-89**.

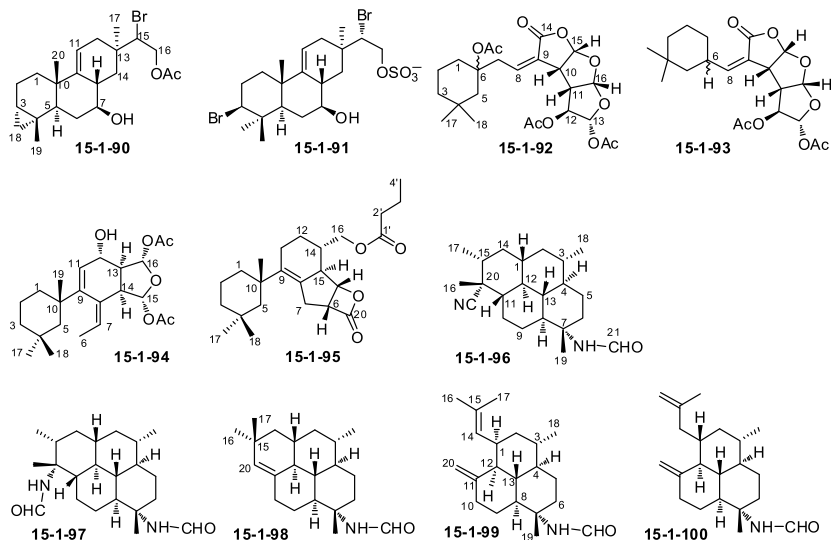
H	15-1-84	15-1-85	15-1-86	15-1-87	15-1-88	15-1-89
1	1.98 m	1.99 m	2.00 m	1.47 m	1.62 m	2.58 dd(9.5, 9.5)
2 α	1.73 m	1.76 m	1.78 m	1.60 m	1.56 m	1.63 m
2 β	1.38 m	1.40 m	1.40 m	1.28 m	1.25 m	1.50 m
3 α	1.65 m	1.66 m	1.66 m	1.66 m	1.63 m	1.98 m
3 β	1.83 m	1.84 m	1.84 m	1.80 m	1.80 m	2.05 m

Table 15-1-29 (continued)

H	15-1-84	15-1-85	15-1-86	15-1-87	15-1-88	15-1-89
4	2.52 m	2.52 m	2.52 m	2.52 m	2.51 m	
5						5.37 dd(10.0, 9.5)
6	2.50 m	2.50 m	2.48 m	2.50 m	2.49 m	α 2.02 m, β 2.35 m
7	2.44 m	2.45 m	2.46 m	2.47 m	2.46 m	α 2.25 m, β 2.01 m
9	2.41 m	2.42 m	2.42 m	2.40 m	2.41 m	2.32 q(9.5)
10 α	2.03 m	2.05 m	1.95 m	1.55 m	1.49 m	2.23 dd(10.0, 9.5)
10 β	1.68 m	1.66 m	1.72 m		1.78 m	1.53 m
12				1.52 m	4.68 dd(9.2, 3.6)	
13	2.35 m	2.35 m	2.38 m	2.31 m	2.16 m	5.87 d(10.5)
14	1.77 m	1.76 m	1.94 m		5.32 dd(7.2, 6.8)	5.95 d(10.5)
15	4.88 m	3.79 m		2.63 sept(7.0)		
16			1.45 s	1.10 d(7.0)	4.51 d(12.0) 4.60 d(12.0)	1.35 s
17	1.23 d(6.0)	1.25 d(6.3)	1.45 s	1.10 d(7.0)	1.73 s	3.59 d(12.5) 3.64 d(12.5)
18	1.18 s	1.20 s	1.21 s	0.95 s	1.01 s	1.11 s
19	4.91 s 4.92 s	4.91 s 4.92 s	4.91 s 4.92 s	4.88 s 4.89 s	4.88 s 4.91 s	4.68 s 5.06 s
20	1.05 d(7.0)	1.05 d(6.9)	1.05 d(7.0)	1.03 d(7.0)	1.00 d(6.8)	1.60 s
OAc	2.05 s		1.99 s		2.07 s, 2.13 s	

Table 15-1-30: Compounds, MFs, and test solvents of marine terpenoids 15-1-90~15-1-100.

No.	Compounds	MFs	Test solvents	References
15-1-90	16-acetoxy-15-bromo-7-hydroxy-9(11)-parguerene	C ₂₂ H ₃₃ BrO ₃	CDCl ₃	[1]
15-1-91	tedanol	C ₂₀ H ₃₁ Br ₂ O ₅ S	CD ₃ OD	[25]
15-1-92	gracilin J	C ₂₄ H ₃₂ O ₁₀	CDCl ₃	[26]
15-1-93	gracilin K	C ₂₁ H ₂₈ O ₈	CDCl ₃	[26]
15-1-94	gracilin L	C ₂₃ H ₃₄ O ₆	CDCl ₃	[26]
15-1-95	3'-norspongiolactone	C ₂₄ H ₃₆ O ₄	CDCl ₃	[26]
15-1-96	(1S,3S,4R,7S,8S,11S,12S,13S,15R,20R)-7-formamido-20-isocyanoisocycloamphilectane	C ₂₂ H ₃₄ N ₂ O	CDCl ₃	[27]
15-1-97	(1S,3S,4R,7S,8S,11S,12S,13S,15R,20R)-7,20-diform-amidoisocycloamphilectane	C ₂₂ H ₃₆ N ₂ O ₂	CDCl ₃	[27]
15-1-98	(1S*,3S*,4R*,7S*,8S*,12S*,13S*)-7-formamidocycloamphilect-11(20)-ene	C ₂₁ H ₃₃ NO	CDCl ₃	[27]
15-1-99	(1R*,3S*,4R*,7S*,8S*,12S*,13S*)-7-formamidoamp-hilecta-11(20),14-diene	C ₂₁ H ₃₃ NO	CDCl ₃	[27]
15-1-100	(1S*,3S*,4R*,7S*,8S*,12S*,13S*)-7-formamidoamp-hilecta-11(20),15-diene	C ₂₁ H ₃₃ NO	CDCl ₃	[27]

**Table 15-1-31:** ^1H NMR spectroscopic data of marine terpenoids **15-1-90** and **15-1-91**.

H	15-1-90	15-1-91	H	15-1-90	15-1-91
1	α 0.85 dt(12.9, 6.0) β 1.59 m	ax 1.55 ddd(13.5, 13.3, 3.6) eq 1.70 ddd(13.3, 3.6, 3.4)	11	5.33 br d(6.2)	5.31 ddd(6.3, 2.0, 1.7)
2	α 1.79 m β 1.96 m	ax 2.27 dddd(13.6, 13.5, 12.7, 3.4) eq 2.16 dddd(13.6, 4.2, 3.6, 3.6)	12	α 1.80 m β 2.42 m	ax 1.82 ddd(17.3, 3.5, 2.0) eq 2.46 ddd(17.3, 6.3, 3.1, 1.6)
3	0.63 dt(9.2, 6.2)	4.02 dd(12.7, 4.2)	14	α 1.38 dd(13.1, 10.3)	ax 1.37 dd(13.9, 10.7)
5	1.12 dd(13.5, 3.4)	1.08 dd(12.7, 2.5)	15	4.27 m	4.37 dd(8.9, 3.4)
6	α 2.11 m β 1.60 m	ax 1.62 ddd(12.7, 12.7, 11.1) eq 1.98 ddd(12.7, 4.7, 2.5)	16	4.55 m, 4.25 m	4.49 dd(11.8, 3.4)
7	3.14 td(10.4, 4.6)	3.02 ddd(11.2, 9.7, 4.7)	17	1.05 s	1.06 s
8	2.33 m	2.37 dddddd(10.7, 9.8, 5.9, 3.5, 1.7, 1.6)	18	0.40 dd(9.3, 4.1) -0.05 dd(5.6, 4.4)	1.13 s
			19	0.97 s	1.01 s
			20	0.96 s	1.08 s

Table 15-1-32: ¹H NMR spectroscopic data of marine terpenoids **15-1-92~15-1-95**.

H	15-1-92	15-1-93	15-1-94	15-1-95
1	2.10 m, 1.20 m	1.66 m, 1.08 m	1.56 m	1.80 m, 0.99 m
2	1.61 m, 1.45 m	1.51 m	1.48 m	1.45 m
3	1.36 m, 1.05 m	1.33 m, 1.17 m	1.36 m, 1.04 m	1.22 m, 1.08 m
5	2.24 m, 1.10 m	1.33 m, 0.97 m	1.45 m, 1.32 m	1.90 m, 1.03 m
6	1.99 s(OAc)	3.72 m	1.62 d(7.0)	3.88 ddd(6.0, 11.2, 16.0)
7	3.53 ddd(1.2, 7.0, 16.0) 3.28 ddd(1.2, 7.0, 16.0)		5.43 q(7.0)	3.02 dd(16.0, 11.0) 2.68 dd(16.0, 6.0)
8	6.53 ddt(1.5, 7.0, 16.0)	6.28 dd(2.0, 10.0)		
10	3.91 ddd(1.5, 6.0, 12.0)	3.83 ddd(1.5, 6.0, 12.0)		
11	3.10 q(6.0)	3.72 q(6.0)	5.92 d(4.0)	2.02 m, 1.78 m
12	5.03 s, 2.05 s(OAc)	5.13 s, 2.07 s(OAc)	4.14 dd(4.0, 5.5)	2.28 m
13	6.19 s, 2.05 s(OAc)	6.17 s, 2.06 s(OAc)	2.70 ddd(2.2, 5.0, 9.0)	1.63 m
14			3.07 dd(1.2, 9.0)	2.39 d(11.0)
15	5.99 d(6.0)	5.98 d(6.0)	6.36 s, 2.05 s(OAc)	4.71 dd(6.0, 3.8)
16	6.12 d(6.0)	6.12 d(6.0)	5.99 d(2.0) 2.07 s(OAc)	4.12 dd(11.2, 6.5) 4.02 dd(11.2, 6.5)
17	0.84 s	0.85 s	0.87 s	0.76 s
18	0.92 s	0.87 s	0.98 s	0.83 s
19			1.24 s	0.95 s
2'				2.26 m, 1.28 m
3'				1.61 m, 0.82 m
4'				0.92 t

Table 15-1-33: ¹H NMR spectroscopic data of marine terpenoids **15-1-96~15-1-99**.

H	15-1-96	15-1-97	15-1-98	15-1-99
1	1.13 m	1.14 m	1.41 m	2.88 m
2	0.82 m, 1.54 m	0.77 m, 1.54 m	0.90 m, 1.60 m	1.27 m, 1.62 m
3	1.12 m	1.12 m	1.26 m	1.27 m
4	0.77 m	0.70 m	1.32 m	<i>cis</i> 0.85 m, <i>trans</i> 0.78 m
5	1.03 m, 1.89 m	1.01 m, 1.78 m	1.04 m, 1.74 m	1.08 m, 1.89 m
6	<i>cis</i> 1.93 m, 2.08 m <i>trans</i> 1.56 m, 1.83 m	<i>cis</i> 1.96 m <i>trans</i> 1.57 m, 1.80 m	<i>cis</i> 1.32 m, 2.04 m <i>trans</i> 1.58 m, 1.86 m	<i>cis</i> 1.93 m, 2.23 m <i>trans</i> 1.56 m, 1.88 m
8	<i>cis</i> 1.62 m <i>trans</i> 1.11 m	<i>cis</i> 1.57 m <i>trans</i> 0.98 m	<i>cis</i> 1.81 m <i>trans</i> 1.15 m	<i>cis</i> 2.03 m <i>trans</i> 1.23 m
9	0.98 m, 1.88 m	0.99 m, 1.86 m	1.04 m, 1.90 m	1.08 m, 1.81 m
10	1.25 m, 1.98 m	1.00 m, 1.89 m	1.95 m, 2.23 m	2.00 m, 2.34 m
11	0.95 m	0.96 m		

Table 15-1-33 (continued)

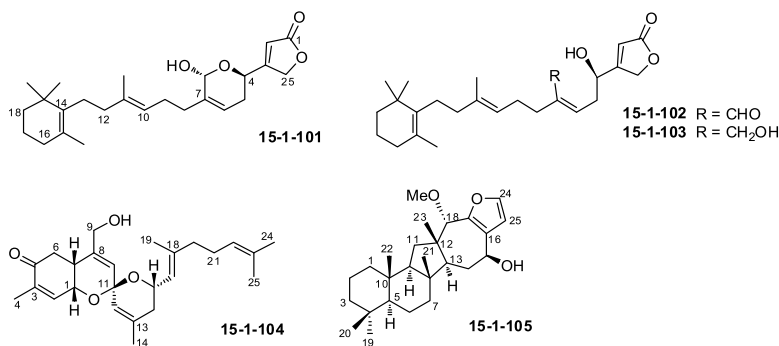
H	15-1-96	15-1-97	15-1-98	15-1-99
12	0.94 m	0.75 m	0.81 m	1.88 m
13	<i>trans</i> 0.77 m	0.70 m	0.79 m	1.31 m
14	1.13 m, 1.41 m	0.90 m, 1.40 m	1.08 m, 1.32 m	5.27 m
15	1.43 m	1.46 m		
16	1.35 s	1.34 s	0.96 s	1.71 s
17	1.02 d(6.4)	0.86 d(6.4)	0.93 s	1.65 s
18	<i>cis</i> 0.86 d(6.4)	0.85 d(6.4)	<i>cis</i> 0.90 d(6.1)	<i>cis</i> 0.82 q(5.7)
	<i>trans</i> 0.88 d(6.4)		<i>trans</i> 0.92 d(6.1)	<i>trans</i> 0.84 q(5.7)
19	<i>cis</i> 1.26 s	<i>cis</i> 1.22 s	<i>cis</i> 1.23 s	<i>cis</i> 1.24 s
	<i>trans</i> 1.21 s	<i>trans</i> 1.17 s	<i>trans</i> 1.20 s	<i>trans</i> 1.25 s
20			<i>cis</i> 5.02 q(2.0)	<i>cis</i> 4.50 q(1.5), 4.68 q(1.5)
			<i>trans</i> 5.04 q(2.0)	<i>trans</i> 4.52 q(1.5), 4.70 q(1.5)
21	<i>cis</i> 8.06 d(2.3)	<i>cis</i> 8.02 d(1.8)	<i>cis</i> 8.06 d(2.0)	<i>cis</i> 8.06 d(2.0)
	<i>trans</i> 8.26 br d(12.4)	<i>trans</i> 8.06 br d(12.1)	<i>trans</i> 8.28 br d(12.2)	<i>trans</i> 8.28 br d(12.4)
22		<i>trans</i> 8.22 br d(12.1)		
NH <i>cis</i>	5.14 br s	5.31 br s	5.10 br s	5.13 br s
NH <i>trans</i>	5.72 br d(12.4)	6.25 br d(12.1)	5.57 br d(12.2)	5.72 br d(12.4)
		5.92 br d(12.1)		

Table 15-1-34: ¹H NMR spectroscopic data of marine terpenoid 15-1-100.

H	15-1-100	H	15-1-100	H	15-1-100
1	1.77 m	12	1.45 m	20	<i>cis</i> 4.80 s, 4.58 s
2	0.65 m, 1.82 m	13	0.75 m		<i>trans</i> 4.83 s, 4.60 s
3	1.06 m	14	1.46 m, 2.61 br d(14.8)	21	<i>cis</i> 8.05 d(2.0)
4	0.92 m	16	4.65 s, 4.74 s		<i>trans</i> 8.27 d(12.2)
5	0.98 m, 1.87 m	17	1.71 s	22	
6	1.56 m, 1.85 m	18	<i>cis</i> 0.86 d(5.6)	NH <i>cis</i>	5.06 br s
8	1.61 m		<i>trans</i> 0.88 d(5.6)	NH <i>trans</i>	5.54 br d(12.2)
9	1.23 m, 1.95 m	19	<i>cis</i> 1.19 s		
10	1.90 m, 2.33 m		<i>trans</i> 1.20 s		

Table 15-1-35: Compounds, MFs, and test solvents of marine terpenoids 15-1-101~15-1-105.

No.	Compounds	MFs	Test solvents	References
15-1-101	deoxymanoalide	C ₂₅ H ₃₆ O ₄	CDCl ₃	[28]
15-1-102	deoxysecomanoalide	C ₂₅ H ₃₆ O ₄	CDCl ₃	[28]
15-1-103	manoalide diol	C ₂₅ H ₃₈ O ₄	CDCl ₃	[28]
15-1-104	phorbaketol A	C ₂₅ H ₃₄ O ₄	CD ₃ OD	[29]
15-1-105	similan A	C ₂₆ H ₄₀ O ₃	CDCl ₃	[30]

**Table 15-1-36:** ¹H NMR spectroscopic data of marine terpenoids 15-1-101~15-1-105.

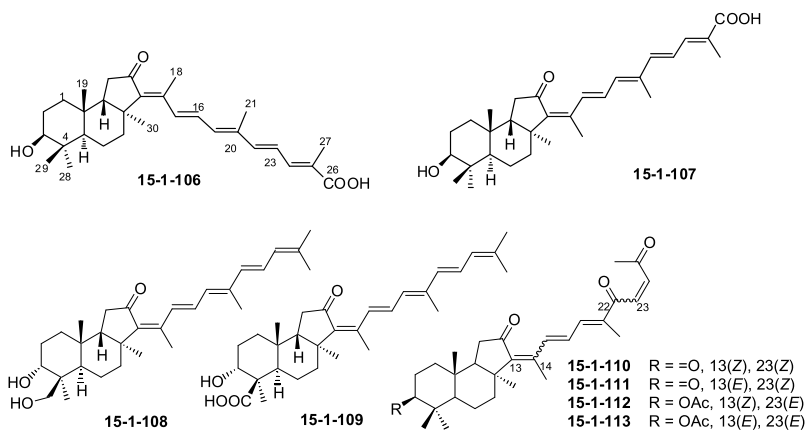
H	15-1-101	15-1-102	15-1-103	15-1-104	15-1-105
1				4.49 dd(5.5, 3.3)	1.39 m, 0.96 m
2	6.03 br q(1.9)	6.04 dt(3.4, 1.9)	5.98 br s	6.68 dd(5.5, 1.2)	1.62 m, 1.41 m
3					1.37 m
					1.18 td(13.9, 4.0)
4	4.94 dd(10.5, 5.4)	4.85 br t(6.4)	4.64 br t(5.5)	1.81 s	
5	2.23 m, 2.17 m	2.80 t(6.7)	2.54 m		0.94 m
6	5.71 m	6.51 t(7.0)	5.39 t(8.0)	2.43 dd(16.1, 13.7)	1.57 m
				2.58 dd(16.1, 3.9)	1.38 m
7				2.59 ddd(13.7, 3.9, 3.4)	1.67 m
					1.11 td(12.5, 3.9)
8	2.16 m	2.32 t(7.5)	2.16 m		
9	2.03 m	2.09 q(7.5)	2.20 m	4.03 d(14.7), 4.07 d(14.7)	1.34 m
10	5.14 br t(6.5)	5.12 br t(7.2)	5.13 m	5.54 br s	
11					1.81 dd(8.6, 3.3)
					1.32 m
12	2.03 m	1.97 m	2.03 m	5.28 br s	
13	2.16 m	2.03 m	2.03 m		1.89 d(11.3)
14				1.75 s	1.83 dd(12.5, 5.6)
					1.64 m
15				1.85 dd(17.6, 3.4) 2.02 dd(17.6, 11.3)	4.66 dd(9.9, 5.3)
16	1.90 t(6.5)	1.90 t(6.3)	1.90 t(6.5)	4.74 ddd(11.3, 8.1, 3.4)	
17	1.56 m	1.57 m	1.57 m	5.22 br d(8.1)	
18	1.42 m	1.41 m	1.41 m		3.85 s, 3.30 s(OMe)
19				1.77 s	0.85 s
20	0.99 s	0.98 s	0.99 s	2.05 m	0.83 s
21	0.99 s	0.98 s	0.99 s	2.13 m	0.78 s

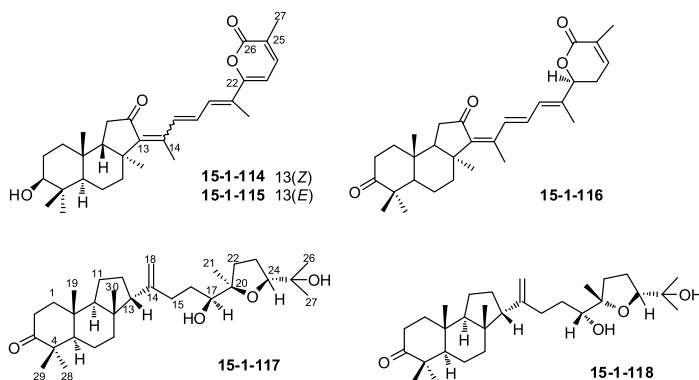
Table 15-1-36 (continued)

H	15-1-101	15-1-102	15-1-103	15-1-104	15-1-105
22	1.60 s	1.59 s	1.60 s	5.12 brt(7.4)	0.83 s
23	1.65 s	1.60 s	1.64 s		0.95 s
24	5.32 br s	9.43 s	4.19 d(11.5) 4.10 d(11.5)	1.68 s	7.30 d(1.8)
25	4.87 dd(17.8, 1.9) 4.92 dd(17.8, 1.9)	4.91 br s	4.88 br s	1.61 s	6.53 br d(1.5)

Table 15-1-37: Compounds, MFs, and test solvents of marine terpenoids 15-1-106~15-1-118.

No.	Compounds	MFs	Test solvents	References
15-1-106	stellettin L	$C_{30}H_{42}O_4$	$CDCl_3$	[31]
15-1-107	stellettin M	$C_{30}H_{42}O_4$	$CDCl_3$	[31]
15-1-108	stellettin J	$C_{30}H_{44}O_3$	$CDCl_3$	[32]
15-1-109	stellettin K	$C_{30}H_{42}O_4$	$CDCl_3$	[32]
15-1-110	isogeoditin A	$C_{29}H_{38}O_4$	C_6D_6	[33]
15-1-111	13-(E)-isogeoditin A	$C_{29}H_{38}O_4$	$CDCl_3$	[33]
15-1-112	isogeoditin B	$C_{31}H_{42}O_5$	C_6D_6	[33]
15-1-113	jaspolide E	$C_{31}H_{42}O_5$	C_6D_6	[34]
15-1-114	jaspolide A	$C_{30}H_{40}O_4$	$CDCl_3$	[34]
15-1-115	jaspolide B	$C_{30}H_{40}O_4$	$CDCl_3$	[34]
15-1-116	22,23-dihydrostellettin B	$C_{30}H_{40}O_4$	$CDCl_3$	[33]
15-1-117	(17S,20R,24R)-17,25-dihydroxy-20,24-epoxy-14(18)-malabaricen-3-one	$C_{30}H_{50}O_4$	$CDCl_3$	[35]
15-1-118	(17R,20S,24R)-17,25-dihydroxy-20,24-epoxy-14(18)-malabaricen-3-one	$C_{30}H_{50}O_4$	$CDCl_3$	[35]



**Table 15-1-38:** ^1H NMR spectroscopic data of marine terpenoids 15-1-106~15-1-110.

<i>H</i>	15-1-106	15-1-107	15-1-108	15-1-109	15-1-110
1	1.49 m, 1.55 m	1.49 m, 1.55 m	1.23 m, 1.85 m	1.14 m, 1.86 m	^① ddd(6.0, 10.0, 12.0) 1.61 ddd(3.5, 10.0, 12.0)
2	1.70 m, 1.83 m	1.70 m, 1.83 m	1.71 m, 1.86 m	1.69 m, 2.20 m	2.40 ddd(6.0, 10.0, 12.0) 2.28 ddd(3.5, 10.0, 12.0)
3	3.32 m	3.32 m	4.09 dd(2.4, 7.8)	4.16 br s	
5	1.73 m	1.68 m	2.23 m	2.46 br d(11.2)	2.04 dd(1.5, 11.5)
6	1.48 m, 1.56 m	1.46 m, 1.54 m	1.42 m, 1.66 m	1.87 m	1.15 m, 1.26 m
7	2.17 m	2.10 m	2.07 m	2.12 m	1.83 m, 1.64 m
9	1.81 m	1.84 m	1.75 m	1.84 m	1.42 dd(7.0, 15.0)
10	2.19 m	2.17 m			
11	2.22 m	2.20 m	2.19 m	2.24 m	1.97 dd(16.5, 15.0) 2.08 dd(7.0, 16.5)
15	6.73 d(15.0)	8.14 d(15.2)	8.05 d(15.3)	8.05 d(15.2)	9.01 d(15.5)
16	7.01 dd (15.0, 11.6)	6.96 dd (15.2, 1.6)	6.98 dd (11.5, 15.3)	6.98 dd (11.2, 15.2)	6.98 dd (11.5, 15.5)
17	6.42 d(11.6)	6.47 d(11.6)	6.28 d(11.5)	6.29 d(11.6)	7.22 d(11.5)
18	2.33 s	2.04 s	2.02 s	2.04 s	1.82 s
19	1.00 s	1.00 s	1.02 s	0.91 s	0.59 s
21	2.04 s	2.00 s	1.96 s	1.96 s	2.21 s
22	6.62 d(15.1)	6.68 d(15.1)	6.23 d(15.2)	6.24 d(15.2)	
23	6.66 dd (15.1, 9.5)	6.58 dd (15.1, 9.5)	6.52 dd (11.1, 15.2)	6.52 dd (11.2, 15.2)	6.08 d(12.0)
24	7.39 d(9.5)	7.39 d(9.5)	5.94 d(11.0)	5.94 d(11.2)	5.78 d(12.0)
26			1.82 s	1.82 s	1.81 s
27	2.02 s	2.02 s	1.83 s	1.83 s	1.04 s

Table 15-1-38 (continued)

H	15-1-106	15-1-107	15-1-108	15-1-109	15-1-110
28	1.04 s	1.05 s	1.10 s	1.33 s	1.17 s
29	0.83 s	0.83 s	3.57 d(10.4) 3.79 d(10.4)		1.05 s
30	1.40 s	1.37 s	1.36 s	1.40 s	

① Typographic errors exist in the literature, not giving the chemical shift of H-1.

Table 15-1-39: ¹H NMR spectroscopic data of marine terpenoids 15-1-111~15-1-114.

H	15-1-111	15-1-112	15-1-113	15-1-114
1	1.55 m, 2.18 m	1.00 m, 1.29 m	1.05 m, 1.30 m	1.42 m, 1.60 m
2	2.76 m, 2.42 m	1.92 m, 1.68 m	1.67 m, 1.96 m	1.70 m, 1.87 m
3		4.83 dd(5.0, 11.5)	4.84 dd(5.0, 12.0)	3.33 dd(5.0, 11.5)
5	2.40 m	1.60 m	1.63 m	1.67 m
6	1.52 m, 1.55 m	1.51 m, 1.52 m	1.48 m, 1.68 m	1.42 m, 1.70 m
7	1.71 m, 1.70 m	1.30 m, 1.53 m	1.97 m, 2.03 m	2.10 m, 2.20 m
9	1.91 m	1.51 m	1.50 dd(7.0, 13.0)	1.85 dd(7.0, 11.0)
11	2.29 m, 2.39 m	2.07 m, 2.11 m	2.13 dd(7.0, 14.0) 2.04 dd(13.0, 14.0)	2.22 d(11.5) 2.13 m
15	6.94 d(15.0)	9.01 d(15.5)	7.02 d(15.5)	8.26 d(15.0)
16	7.09 dd(15.0, 11.0)	6.96 dd(15.5, 11.5)	6.93 dd(11.0, 15.5)	6.93 dd(11.5, 15.0)
17	7.06 d(11.0)	7.22 d(11.5)	7.32 d(11.0)	7.25 d(11.4)
18	2.37 s	1.78 s	2.63 s	2.06 s
19	0.88 s	0.78 s	0.75 s	1.02 s
21	2.09 s	2.21 s	2.04 s	2.04 s
23	6.75 d(12.0)	6.07 d(12.0)	7.57 d(15.5)	6.23 d(7.0)
24	6.49 d(12.0)	5.77 d(12.0)	7.06 d(15.5)	7.16 d(7.0)
26	2.30 s	1.83 s	1.71 s	
27	1.15 s	1.00 s	1.03 s	2.13 s
28	1.08 s	1.05 s	0.98 s	0.84 s
29	1.46 s	1.07 s	1.11 s	1.06 s
30				1.38 s
OAc		1.89 s	1.88 s	

Table 15-1-40: ¹H NMR spectroscopic data of marine terpenoids 15-1-115~15-1-118.

H	15-1-115	15-1-116	15-1-117	15-1-118
1	1.43 m 1.60 m	1.04 m 1.29 m	ax 1.51 m eq 1.78 ddd(13.4, 7.6, 3.5)	ax – eq 1.78 ddd(13.3, 7.6, 3.5)
2	1.74 m	2.39 m	ax 2.55 ddd(16.1, 10.9, 7.6)	ax 2.56 ddd(16.1, 10.9, 7.6)

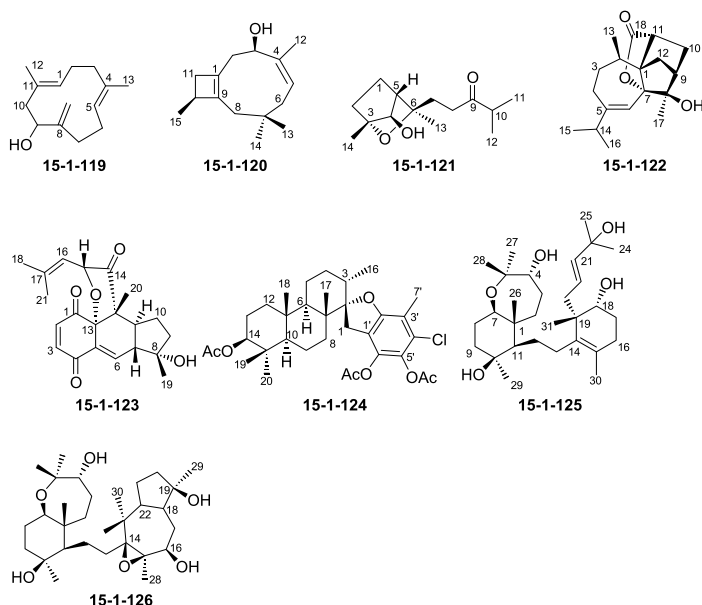
Table 15-1-40 (continued)

H	15-1-115	15-1-116	15-1-117	15-1-118
	1.82 m	2.74 m	eq 2.39 ddd(16.1, 7.3, 3.5)	eq 2.39 ddd(16.1, 7.3, 3.5)
3	3.37 dd(4.9, 11.5)			
5	1.77 m	2.37 m	1.26 m	—
6	1.44 m, 1.68 m	1.24 m, 1.52 m	1.53 m	—
7	1.90 m, 2.00 m	2.23 m	ax 1.15 m, eq 1.63 m	—
9	1.89 dd(6.8, 11.5)	1.90 m	1.40 dd(7.7, 12.7)	—
11	2.32 m	2.18 m 2.25 m	1.51 m	—
12			α 1.59 m, β 2.01 m	—
13			2.18 m	—
15	6.96 d(15.3)	8.08 d(15.0)	2.13 m, 2.18 m	—
16	6.98 dd(11.4, 15.3)	6.85 dd(11.0, 15.0)	1.43 m, 1.49 m	—
17	7.28 d(11.4)	6.33 d(11.0)	3.52 dd(10.2, 2.0)	3.57 dd(10.2, 2.2)
18	2.34 s	2.07 s	4.62 br s 4.91 q(1.2, 1.2, 1.2)	4.62 br s 4.91 q(1.2, 1.2, 1.2)
19	1.01 s	0.88 s	0.98 s	0.98 s
21	2.04 s	1.93 s	1.14 s	1.15 ^①
22		4.87 dd(3.5, 12.5)	1.56 m, 2.10 m	—
23	6.26 d(6.9)	2.60 m	1.87 m	—
24	7.20 d(6.9)	6.65 br s	3.77 dd(5.4, 10.6)	3.83 t(7.4, 7.4)
26			1.13 s	1.17 ^①
27	2.15 s	1.97 s	1.22 s	1.27 ^①
28	1.09 s	1.08 s	1.07 s	1.07 s
29	1.09 s	1.14 s	1.04 s	1.04 s
30	1.43 s	1.41 s	1.03 s	1.02 s

^① Assignments may be exchanged.

Table 15-1-41: Compounds, MFs, and test solvents of marine terpenoids 15-1-119~15-1-126.

No.	Compounds	MFs	Test solvents	References
15-1-119	gibberosin N	C ₁₄ H ₂₂ O	CDCl ₃	[24]
15-1-120	capillosanol	C ₁₅ H ₂₄ O	CD ₃ COCD ₃	[36]
15-1-121	chabranol	C ₁₄ H ₂₄ O ₃	CDCl ₃	[36]
15-1-122	nanolobatolide	C ₁₈ H ₂₆ O ₃	CDCl ₃	[37]
15-1-123	rossinone B	C ₂₁ H ₂₄ O ₅	CDCl ₃	[38]
15-1-124	4'-chlorostypotriol triacetate	C ₃₃ H ₄₅ ClO ₇	CDCl ₃	[39]
15-1-125	siphonellinol C	C ₃₀ H ₅₂ O ₅	CDCl ₃	[40]
15-1-126	sipholenol I	C ₃₀ H ₅₂ O ₆	CDCl ₃	[40]

**Table 15-1-42:** ^1H NMR spectroscopic data of marine terpenoids **15-1-119**~**15-1-122**.

H	15-1-119	15-1-120	15-1-121	15-1-122
1	4.84 dd(10.0, 4.4)		α 1.84 m, β 2.64 m	
2	2.02~2.14 m	1.76 m, 1.54 m	α 1.44 m, β 1.62 m	1.97 m
3	1.98~2.03 m	4.77 br s, 3.78 d(4.0, OH)		α 1.42 m, β 1.78 m
4			4.13 br s	α 2.52 m, β 2.02 m
5	5.18 dd(8.4, 7.2)	5.59 br s	2.00 br s	
6	2.24 m	2.31 d(15.5), 2.02 d(15.5)		5.33 s
7	1.76~2.35 m		1.91 m, 1.57 m	
8		1.92 d(15.0), 2.20 d(15.0)	2.35 m, 2.63 m	
9	4.14 dd(10.4, 4.4)			2.28 d(3.5)
10	α 2.02 m β 2.56 dd(12.0, 4.0)	2.25 m	2.58 m	α 1.86 d(10.5) β 2.04 m
11		α 1.82 d(13.0), β 1.40 d(13.0)	1.09 d(7.2)	2.64 d(10.5)
12	1.59 s	1.68 s	1.11 d(7.2)	α 2.42 d(11.0) β 1.46 d(11.0)
13	1.49 s	1.02 s	1.16 s	0.93 d(6.5)
14	5.09 s, 5.29 s	1.03 s	1.20 s	2.37 m
15		0.97 d(7.0)		1.04 d(7.0)
16				1.05 d(7.0)
17				1.28 s

Table 15-1-43: ^1H NMR spectroscopic data of marine terpenoids **15-1-123** and **15-1-124**.

H	15-1-123	15-1-124	H	15-1-123	15-1-124
1		3.10 d(16.4), 2.68 d(16.4)	13		1.31 m, 1.58 t(13.1, 3.3)
2	6.79 d(10.5)		14		4.51 dd(12.0, 5.0)
3	6.90 d(10.5)	1.38 m	15	4.55 d(8.8)	
4		1.46 m, 1.15 m	16	5.07 dm(8.9)	0.72 d(6.4)
5		1.68 m, 1.41 m	17		0.93 s
6	7.44 d(1.9)	1.52 d(12.6)	18	1.76 d(1.1)	0.87 s
7	2.06 dd(12.4, 1.9)		19	1.54 s	0.83 s
8		1.01 dd(15.1, 6.6)	20	1.10 s	0.84 s
9	1.92 m	1.47 m, 1.12 m	21	1.73 d(1.2)	
10	1.53 m	0.86 m	7'		2.25 s
11	2.67 m		OAc		2.10 s, 2.31 s, 2.26 s
12		1.75 t(13.1, 3.3), 1.45 t(13.1)			

Table 15-1-44: ^1H NMR spectroscopic data of marine terpenoids **15-1-125** and **15-1-126**.

H	15-1-125	15-1-126	H	15-1-125	15-1-126
2	1.38 m, 1.52 m	1.46 m, 1.64 m	20	2.13 dd(14.6, 6.2)	1.36 m, 1.60 m
3	1.74 m, 1.98 m	1.68 m, 2.02 m		2.27 dd(14.6, 8.1)	
4	3.81 d(6.6)	3.79 d(6.6)	21	5.46 ddd(15.8, 8.1, 6.2)	1.21 m, 1.79 m
7	3.52 dd(12.1, 4.4)	3.48 dd(11.7, 4.4)	22	5.63 d(15.8)	2.48 d(4.4)
8	1.38 m, 1.71 m	1.37 m, 1.74 m	24	1.28 s	1.03 s
9	1.60 m	1.46 m, 1.64 m	25	1.28 s	1.25 s
11	0.88 m	0.83 m	26	0.98 s	1.12 s
12	1.46 m	1.55 m, 1.63 m	27	1.26 s	1.15 s
13	2.04 m	1.50 m	28	1.12 s	1.42 s
		2.22 ddd(14.3, 12.4, 7.3)	29	1.23 s	1.14 s
16	2.02 m	3.99 dd(9.9, 6.6)	30	1.67 s	1.00 s
17	1.70 m	1.28 m, 2.01 m	31	1.06 s	1.24 s
18	3.60 dd(9.6, 2.9)	1.84 br m			

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15.2 Marine steroids

15.2.1 Marine pregnane-type steroids

Table 15-2-1: Compounds, MFs, and test solvents of marine pregnane-type steroids 15-2-1~15-2-11.

No.	Compounds	MFs	Test solvents	References
15-2-1	ximaasteroid B	C ₂₅ H ₃₈ O ₄	CDCl ₃	[41]
15-2-2	ximaasteroid C	C ₂₅ H ₃₆ O ₄	CDCl ₃	[41]
15-2-3	stereonsteroid A	C ₂₁ H ₃₄ O ₂	CDCl ₃	[42]
15-2-4	stereonsteroid B	C ₂₃ H ₃₄ O ₃	CDCl ₃	[42]
15-2-5	stereonsteroid C	C ₂₉ H ₄₆ O ₇	CDCl ₃	[42]
15-2-6	stereonsteroid D	C ₃₁ H ₄₈ O ₈	CDCl ₃	[42]
15-2-7	stereonsteroid E	C ₂₉ H ₄₄ O ₇	CDCl ₃	[42]
15-2-8	stereonsteroid F	C ₂₉ H ₄₆ O ₆	CDCl ₃	[42]
15-2-9	stereonsteroid G	C ₂₉ H ₄₆ O ₆	CDCl ₃	[42]
15-2-10	stereonsteroid H	C ₂₉ H ₄₄ O ₆	CDCl ₃	[42]
15-2-11	stereonsteroid I	C ₂₆ H ₄₂ O ₅	CDCl ₃	[42]

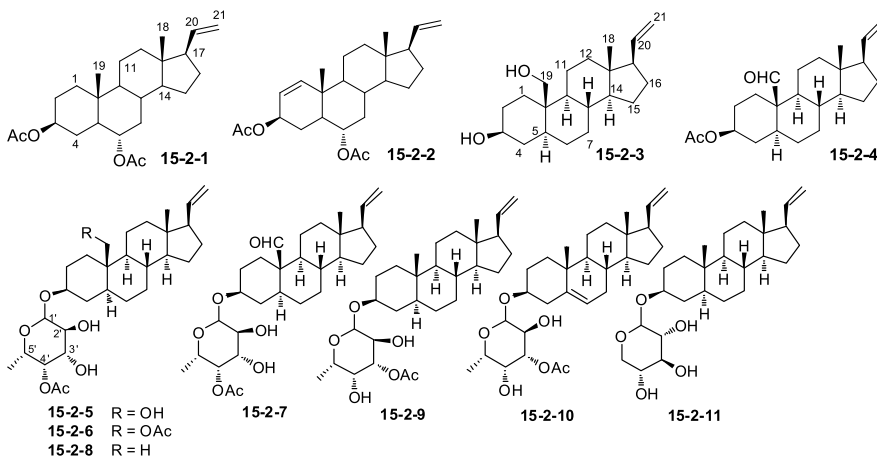


Table 15-2-2: ¹H NMR spectroscopic data of marine pregnane-type steroids 15-2-1 and 15-2-2.

H	15-2-1	15-2-2	H	15-2-1	15-2-2
1	α 1.06 m, β 1.72 m	5.98 d(10.4)	12	α 1.05 m, β 1.67 m	α 1.02 m, β 1.70 m
2	α 1.86 m, β 1.53 m	5.46 br d(10.4)	14	1.08 m	1.10 m
3	4.67 m	5.35 br t(7.6)	15	α 1.62 m, β 1.18 m	α 1.62 m, β 1.20 m
	2.03 s(OAc)	2.06 s(OAc)	16	α 1.77 m, β 1.53 m	α 1.80 m, β 1.56 m
4	α 1.88 m, β 1.28 m	α 2.07 m, β 1.47 m	17	1.97 m	1.97 m
5	1.30 m	1.60 m	18	0.58 s	0.59 s
6	4.67 m	4.77 dt(11.3, 4.7)	19	0.90 s	0.99 s
	2.02 s(OAc)	2.05 s(OAc)	20	5.75 ddd	5.73 ddd
7	α 0.93 m, β 2.03 m	α 0.97 m, β 2.03 m		(16.7, 10.7, 7.6)	(17.3, 10.3, 6.0)
8	1.52 m	1.53 m	21	4.97 d(16.7)	4.97 d(17.3)
9	0.74 dt(11.8, 4.1)	0.92 m		4.96 d(10.7)	4.96 d(10.3)
11	α 1.53 m, β 1.26 m	α 1.72 m, β 1.35 m			

Table 15-2-3: ^1H NMR spectroscopic data of marine pregnane-type steroids **15-2-3~15-2-5**.

H	15-2-3	15-2-4	15-2-5
1	0.85 m, 2.28 dt(13.5, 3.3)	0.99 m, 2.43 dt(13.2, 3.3)	0.86 m, 2.27 dt(13.2, 3.2)
2	1.40 m, 1.90 m	1.36 m, 1.93 m	1.39 m, 1.45 m
3	3.65 m	4.72 m	3.60 m
4	1.01 m, 1.74 m	1.50 m, 1.80 m	0.99 m, 1.72 m
5	1.24 m	1.45 m	1.22 m
6	1.17 m, 1.28 m	1.54 m, 1.78 m	1.55 m, 1.70 m
7	1.18 m, 1.26 m	1.11 m, 1.81 m	0.90 m, 1.76 m
8	1.53 m	1.45 m	1.54 m
9	0.74 m	0.97 m	0.72 m
11	1.57 m, 1.72 m	1.24 m, 1.72 m	1.56 m, 1.68 m
12	1.39 m, 1.70 m	1.02 m, 1.68 m	1.39 m, 1.75 m
14	1.04 m	0.98 m	1.15 m
15	1.73 m	1.20 m, 1.72 m	1.21 m, 1.62 m
16	1.58 m, 1.80 m	1.59 m, 1.82 m	1.56 m, 1.81 m
17	1.97 m	1.97 m	1.96 m
18	0.64 s	0.53 s	0.61 s
19	3.81 d(11.4), 3.95 d(11.4)	10.03 s	3.77 d(12.0), 3.90 d(12.0)
20	5.78 ddd(16.2, 10.5, 7.8)	5.73 ddd(17.1, 10.5, 7.8)	5.74 ddd(16.5, 10.8, 7.8)
21	4.97 d(16.2), 4.98 d(10.5)	4.96 d(17.1), 4.97 d(10.5)	4.94 d(16.5), 4.95 d(10.8)
1'			5.02 d(3.6)
2'			3.74 dd(9.6, 3.6)
3'			3.92 dd(9.6, 3.0)
4'			5.16 br d(3.0)
5'			4.09 br q(6.3)
6'			1.10 d(6.3)
OA _c			2.15 s

Table 15-2-4: ^1H NMR spectroscopic data of marine pregnane-type steroids **15-2-6~15-2-8**.

H	15-2-6	15-2-7	15-2-8
1	0.98 m, 2.23 m	0.96 m, 2.42 m	1.08 m, 1.74 m
2	1.41 m, 1.88 m	1.40 m, 1.85 m	1.55 m, 1.86 m
3	3.60 m	3.59 m	3.56 m
4	1.00 m, 1.73 m	1.44 m, 1.88 m	1.32 m, 1.68 m
5	1.32 m	1.37 m	1.12 m
6	1.33 m, 1.89 m	1.38 m, 1.97 m	1.35 m, 1.84 m
7	0.93 m, 2.16 m	1.12 m, 1.93 m	0.96 m, 1.77 m
8	1.53 m	1.70 m	1.42 m
9	0.77 m	0.98 m	0.68 m
11	1.38 m, 1.67 m	1.31 m, 1.75 m	1.36 m, 1.60 m
12	1.34 m, 1.76 m	1.02 m, 2.37 m	1.04 m, 1.72 m
14	1.12 m	0.97 m	1.04 m
15	1.21 m, 1.70 m	1.23 m, 1.75 m	1.24 m, 1.72 m

Table 15-2-4 (continued)

H	15-2-6	15-2-7	15-2-8
16	1.57 m, 1.84 m	1.57 m, 1.80 m	1.62 m, 1.86 m
17	1.97 m	1.98 m	1.95 m
18	0.58 s	0.53 s	0.59 s
19	4.22 d(12.3), 4.35 d(12.3)	10.03 s	0.82 s
20	5.74 ddd(17.1, 10.2, 7.6)	5.74 ddd(17.1, 10.3, 7.8)	5.76 ddd(16.2, 11.1, 7.8)
21	4.96 d(17.1), 4.97 d(10.2)	4.96 d(17.1), 4.97 d(10.3)	4.96 d(16.2), 4.97 d(11.1)
1'	5.04 d(3.6)	5.01 m	5.04 d(3.6)
2'	3.73 dd(9.6, 3.6)	3.72 dd(9.6, 3.6)	3.74 dd(10.8, 3.6)
3'	3.91 dd(9.6, 3.0)	3.88 dd(9.6, 3.0)	3.92 dd(10.8, 3.0)
4'	5.21 br d(3.0)	5.20 br d(3.0)	5.21 br d(3.0)
5'	4.11 br q(6.3)	4.08 br q(6.3)	4.12 br q(6.3)
6'	1.14 d(6.3)	1.13 d(6.3)	1.14 d(6.3)
OAc	2.18 s, 2.07 s	2.17 s	2.18 s

Table 15-2-5: ¹H NMR spectroscopic data of marine pregnane-type steroids **15-2-9~15-2-11**.

H	15-2-9	15-2-10	15-2-11
1	1.03 m, 1.68 m	1.05 m, 1.72 m	0.92 m, 1.65 m
2	1.52 m, 1.84 m	1.62 m, 1.94 m	1.68 m, 2.08 m
3	3.58 m	3.52 m	3.90 m
4	1.29 m, 1.63 m	1.72 m, 1.89 m	1.37 m, 1.81 m
5	1.14 m		0.99 m
6	1.30 m	5.37 d(5.1)	1.16 m
7	0.92 m, 1.73 m	2.02 m	0.82 m, 1.55 m
8	1.36 m	1.56 m	1.21 m
9	0.68 m	0.96 m	0.53 m
11	1.28 m, 1.55 m	1.50 m, 1.58 m	1.15 m, 1.46 m
12	1.02 m, 1.76 m	1.13 m, 1.92 m	0.92 m, 1.61 m
14	1.01 m	1.03 m	0.87 m
15	1.06 m, 1.66 m	1.23 m, 1.75 m	1.18 m, 1.60 m
16	1.56 m, 1.78 m	1.60 m, 1.78 m	1.53 m, 1.64 m
17	1.96 m	1.96 m	1.93 m
18	0.59 s	0.62 s	0.45 s
19	0.83 s	1.03 s	0.66 s
20	5.76 ddd(16.8, 10.2, 8.1)	5.78 ddd(15.9, 11.4, 7.8)	5.74 ddd(15.6, 11.4, 7.8)
21	4.96 d(16.8), 4.97 d(10.2)	4.97 d(11.4)	4.98 m
1'	5.03 d(4.2)	5.05 d(4.2)	4.90 d(8.7)
2'	3.91 dt(4.2, 11.4)	3.93 dt(4.2, 11.4)	4.00 t(8.7)
3'	5.05 dd(10.5, 3.0)	5.07 dd(10.5, 3.0)	4.19 t(8.7)
4'	3.84 br s	3.85 br s	4.25 ddd(11.1, 8.7, 4.8)
5'	4.11 q(6.3)	4.12 q(6.6)	3.76 t(11.1)
			4.39 dd(11.1, 4.8)
6'	1.26 d(6.3)	1.26 d(6.6)	
OAc	2.18 s	2.19 s	

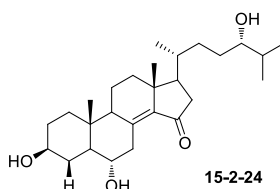
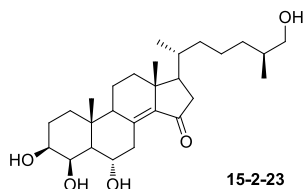
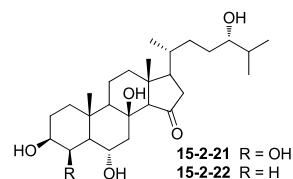
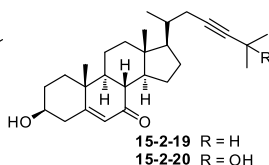
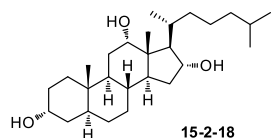
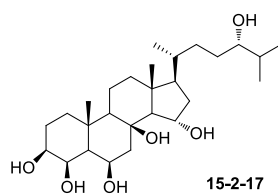
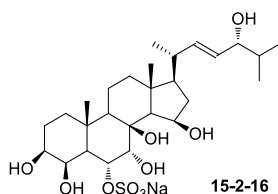
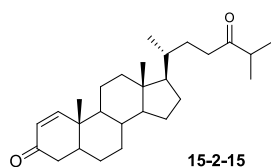
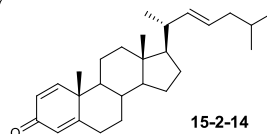
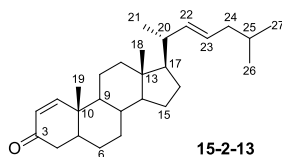
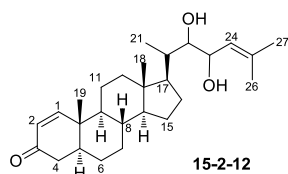
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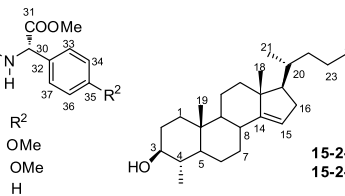
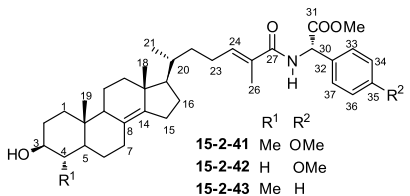
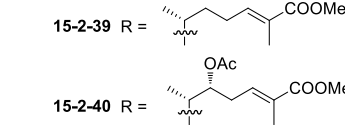
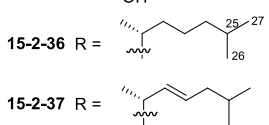
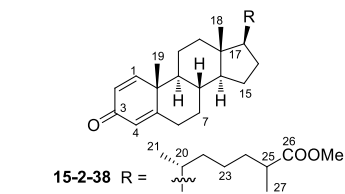
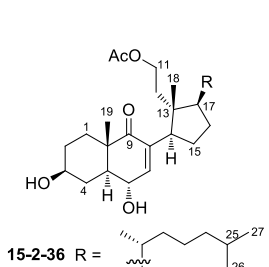
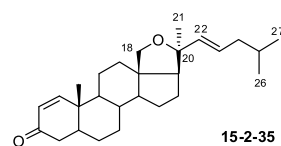
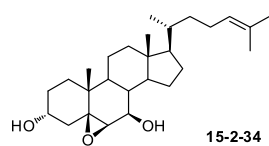
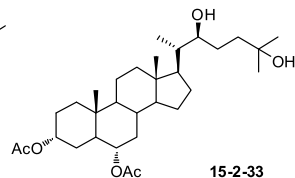
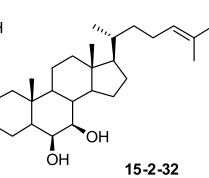
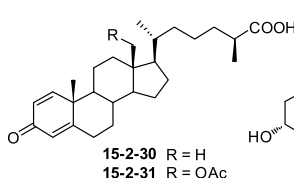
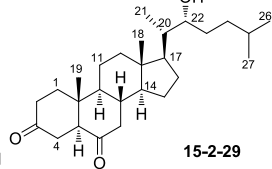
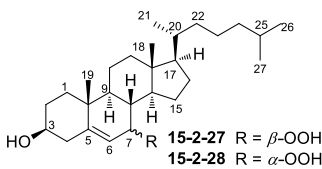
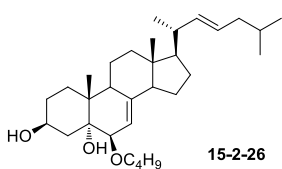
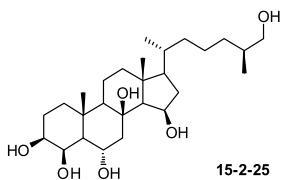
Table 15-2-6: Compounds, MFs, and test solvents of marine cholestane-type steroids 15-2-12~15-2-53.

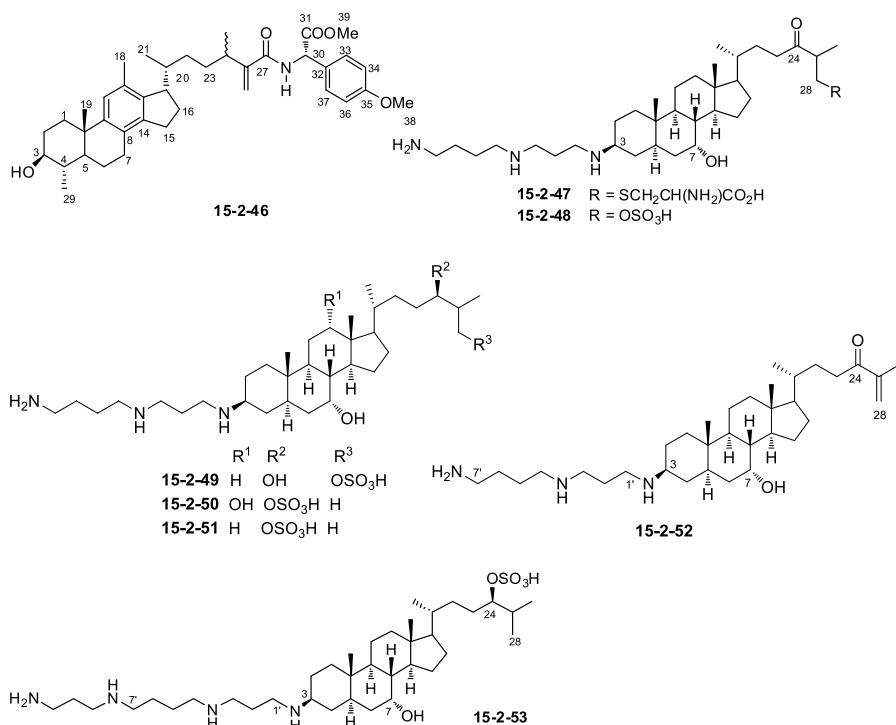
No.	Compounds	MFs	Test solvents	References
15-2-12	22,23-dihydroxycholesta-1,24-dien-3-one	C ₂₇ H ₄₂ O ₃	CDCl ₃	[53]
15-2-13	dendronesterone A	C ₂₇ H ₄₂ O	CDCl ₃	[43]
15-2-14	dendronesterone C	C ₂₇ H ₄₀ O	CDCl ₃	[43]
15-2-15	—	C ₂₇ H ₄₂ O ₂	CDCl ₃	[43]
15-2-16	—	C ₂₇ H ₄₅ NaO ₁₀ S	CD ₃ OD	[44]
15-2-17	—	C ₂₇ H ₄₈ O ₆	CD ₃ OD	[44]
15-2-18	3 α ,12 α ,16 α -trihydroxycholestane	C ₂₇ H ₄₈ O ₃	CDCl ₃	[45]
15-2-19	gelliusterol C	C ₂₇ H ₄₀ O ₂	CDCl ₃	[46]
15-2-20	gelliusterol D	C ₂₇ H ₄₀ O ₃	CDCl ₃	[46]
15-2-21	certonardosterol Q ₂	C ₂₇ H ₄₆ O ₆	CD ₃ OD	[47]
15-2-22	certonardosterol Q ₃	C ₂₇ H ₄₆ O ₅	CD ₃ OD	[47]
15-2-23	certonardosterol Q ₄	C ₂₇ H ₄₄ O ₅	CD ₃ OD	[47]
15-2-24	certonardosterol Q ₆	C ₂₇ H ₄₄ O ₄	CD ₃ OD	[47]
15-2-25	certonardosterol A ₃	C ₂₇ H ₄₈ O ₆	CD ₃ OD	[47]
15-2-26	homaxisterol A ₁	C ₃₁ H ₅₂ O ₃	CD ₃ OD	[48]
15-2-27	7 β -hydroperoxycholesterol	C ₂₈ H ₄₆ O ₃	CDCl ₃	[49]
15-2-28	7 α -hydroperoxycholesterol	C ₂₈ H ₄₆ O ₃	CDCl ₃	[49]
15-2-29	3-keto-22- <i>epi</i> -28- <i>nor</i> -cathasterone	C ₂₇ H ₄₄ O ₃	CDCl ₃	[50]
15-2-30	(25S)-3-oxocholesta-1,4-dien-26-oic acid	C ₂₇ H ₄₀ O ₃	CDCl ₃	[51]
15-2-31	(25S)-18-acetoxy-3-oxocholesta-1,4-dien-26-oic acid	C ₂₉ H ₄₂ O ₅	CDCl ₃	[51]
15-2-32	—	C ₂₇ H ₄₆ O ₃	CDCl ₃	[52]
15-2-33	—	C ₃₁ H ₅₂ O ₆	CDCl ₃	[52]
15-2-34	—	C ₂₇ H ₄₄ O ₃	CDCl ₃	[52]
15-2-35	ximaasteroid A	C ₂₇ H ₄₀ O ₂	CDCl ₃	[41]
15-2-36	11-acetoxy-3 β ,6 α -dihydroxy-9,11- <i>seco</i> -5 α -cholest-7-en-9-one	C ₂₉ H ₄₈ O ₅	CDCl ₃	[54]
15-2-37	—	C ₂₉ H ₄₆ O ₅	CDCl ₃	[52]
15-2-38	methyl-3-oxocholesta-1,4-dien-26-oate	C ₂₈ H ₄₂ O ₃	CDCl ₃	[55]
15-2-39	methyl (24 <i>E</i>)-3-oxocholesta-1,4,24-trien-26-oate	C ₂₈ H ₄₀ O ₃	CDCl ₃	[55]
15-2-40	methyl (22 <i>R</i> ,24 <i>E</i>)-22-acetoxy-3-oxocholesta-1,4,24-trien-26-oate	C ₃₀ H ₄₂ O ₅	CDCl ₃	[55]
15-2-41	polymastiamide C methyl ester	C ₃₈ H ₅₅ NO ₅	CDCl ₃	[56]
15-2-42	polymastiamide D methyl ester	C ₃₇ H ₅₃ NO ₅	CDCl ₃	[56]
15-2-43	polymastiamide F methyl ester	C ₃₇ H ₅₃ NO ₄	CDCl ₃	[56]
15-2-44	—	C ₃₈ H ₅₅ NO ₅	CDCl ₃	[56]
15-2-45	—	C ₃₇ H ₅₃ NO ₄	CDCl ₃	[56]
15-2-46	—	C ₃₉ H ₅₃ NO ₅	CDCl ₃	[56]
15-2-47	—	C ₃₇ H ₆₈ N ₄ O ₄ S	CD ₃ OD	[57]

Table 15-2-6 (continued)

No.	Compounds	MFs	Test solvents	References
15-2-48	—	$C_{34}H_{63}N_3O_6S$	CD_3OD	[57]
15-2-49	—	$C_{34}H_{65}N_3O_6S$	CD_3OD	[57]
15-2-50	—	$C_{34}H_{65}N_3O_6S$	CD_3OD	[57]
15-2-51	squalamine	$C_{34}H_{65}N_3O_5S$	CD_3OD	[57]
15-2-52	—	$C_{34}H_{61}N_3O_2$	CD_3OD	[57]
15-2-53	—	$C_{37}H_{72}N_4O_5S$	CD_3OD	[57]





**Table 15-2-7:** ^1H NMR spectroscopic data of marine cholestane-type steroids 15-2-12~15-2-14.

H	15-2-12	15-2-13	15-2-14
1	7.11 d(10)	7.15 d(9.9)	7.06 d(10.2)
2	5.83 d(10)	5.85 d(9.9)	6.23 dd(10.2, 1.8)
4 α	2.20 m	2.21 dd(18.0, 4.5)	6.07 s
4 β	2.34 m	2.37 dd(18.0, 14.0)	
5	1.91 m	1.96 m	
6	1.42 m	α 1.29 m β 1.72 m	α 2.37 tt(12.9, 2.4) β 2.46 tdd(12.9, 3.9, 1.5)
7	α 1.42 m, β 1.72 m	1.76 m	1.14 m
8	1.43 m	1.50 m	1.28 m
9	0.97 m	1.01 m	1.14 m
11 α	1.72 m,	1.79 m	1.12 m
11 β	1.41 m	1.52 m	1.74 m
12 α	1.24 m	1.18 m	1.18 m
12 β	1.98 m	2.05 m	2.05 m
14	1.09 m	1.18 m	1.01 m
15	α 1.61 m, β 1.10 m	1.60 m	1.61 m
16	α 1.23 m, β 1.90 m	1.46 m	1.72 m

Table 15-2-7 (continued)

H	15-2-12	15-2-13	15-2-14
17	1.52 m	1.22 m	1.15 m
18	0.66 s	0.71 s	0.75 s
19	0.99 s	1.01 s	1.23 s
20	1.47 m	2.07 m	2.00 m
21	0.88 d(6.6)	1.01 d(6.6)	1.00 d(6.6)
22	3.45 br d(8.7)	5.20 dd(15.3, 7.5)	5.19 dd(15.3, 7.8)
23	4.25 t(8.7)	5.27 dt(15.3, 8.4)	5.27 dt(15.3, 8.4)
24	5.03 d(8.7)	1.85 m	1.86 m
25		1.56 m	1.28 m
26	1.73 s	0.86 d(6.6)	0.85 d(6.6)
27	1.75 s	0.87 d(6.6)	0.86 d(6.6)

Table 15-2-8: ^1H NMR spectroscopic data of marine cholestane-type steroids 15-2-15~15-2-17.

H	15-2-15	15-2-16	15-2-17
1	7.14 d(10.0)	1.71 m, 1.01 m	1.72 m, 1.00 m
2	5.86 dd(10.0, 0.5)	1.83 m, 1.57 m	1.88 m, 1.62 m
3		3.47 ddd(3.8, 4.9, 11.9)	3.49 ddd(3.9, 5.2, 11.9)
4	α 2.22 ddd(17.5, 3.5, 0.5) β 2.37 dd(17.5, 14.0)	4.21 br s	4.05 br s
5	1.92 m	1.72 dd(2.3, 12.2)	1.23 m
6	α 1.42 m	5.04 dd(2.8, 12.0)	4.24 m
7	0.95 m, 1.72 m	4.23 d(2.9)	1.59 dd(2.9, 14.9), 2.40 dd(3.0, 15.0)
8	1.43 m		
9	1.22 m	1.09 m	0.97 m
11	α 1.75 m, β 1.45 m	1.82 m, 1.43 m	1.81 m, 1.47 m
12	α 1.31 m, β 1.99 dt(12.5, 3.0)	1.95 m, 1.15 m	1.96 m, 1.23 m
14	1.10 m	1.42 d(5.8)	1.17 d(9.7)
15	1.12 m, 1.59 m	4.48 ddd(2.1, 5.7, 7.7)	4.27 dt(3.2, 9.5)
16	1.12 m, 1.65 m	2.23 m, 1.37 m	1.91 m, 1.69 m
17	1.63 m	1.01 m	1.34 m
18	0.73 s	1.28 s	0.95 s
19	1.01 s	1.22 s	1.42 s
20	2.53 dq(8.5, 7.0)	2.20 m	1.36 m
21	1.03 d(7.0)	1.02 d(6.6)	0.91 d(6.0)
22		5.42 dd(8.0, 15.2)	1.60 m, 0.99 m
23	2.36 m, 2.45 m	5.37 dd(6.8, 15.5)	1.55 m, 1.21 m
24	1.43 m	3.67 t(6.5)	3.19 m
25	1.52 m	1.65 m	1.61 m
26	0.90 d(6.9)	0.90 d(6.7)	0.90 d(6.7)
27	0.90 d(6.9)	0.86 d(6.7)	0.88 d(6.7)

Table 15-2-9: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-18~15-2-22**.

H	15-2-18	15-2-19	15-2-20	15-2-21	15-2-22
1	1.35	1.20 m, 1.96 m	1.20 m, 1.94 m	1.71 dt(13.5, 3.5) 1.00 m	1.73 dt(13.0, 3.3) 0.99 m
2	1.61, 1.38	1.63 m, 1.93 m	1.60 m, 1.94 m	1.81 m, 1.54 m	1.72 m, 1.48 m
3	4.02 m	3.68 m	3.68 m	3.42 ddd (12.0, 5.0, 3.5)	3.47 m
4	1.45, 1.35	2.43 m, 2.50 m	2.40 m, 2.51 ddd (1.9, 4.4, 13.6)	4.24 br s	1.22 m, 2.18 m
5	1.50			0.93 dd(11.0, 2.3)	1.03 m
6	1.17	5.69 d(1.5)	5.70 d(1.6)	4.10 td(11.0, 4.5)	3.64 td(10.5, 4.0)
7	1.61 1.02			3.07 dd(13.0, 4.0) 1.21 dd(13.0, 11.0)	3.00 dd(13.0, 3.5) 1.19 dd(13.0, 11.0)
8	1.39 m	2.24 m	2.24 dd (10.8, 12.2)		
9	1.20	1.49 m	1.36 m	0.82 dd(12.0, 2.3)	0.84 td(12.0, 2.5)
11	1.64	1.56 m	1.58 m	1.71 m, 1.47 m	1.75 m, 1.52 m
12	3.90 t(2.9)	1.14 m 2.02 dt(3.0, 12.9)	1.14 m, 2.02 m	2.14 dt(12.5, 3.3) 1.43 m	2.15 dt(12.3, 3.5) 1.43 m
14	1.84 m	1.36 m	1.34 m	2.08 s	2.10 s
15	1.57 m	2.41 m	2.44 m		
16	3.97 m	1.25 m, 1.93 m	1.88 m	2.42 dd(19.0, 8.0) 1.80 dd(19.0, 9.5)	2.43 dd(18.8, 8.8) 1.80 dd(18.8, 9.8)
17	1.58 m	1.20 m	1.18 m	1.58 m	1.56 m
18	0.70 s	0.69 s	0.70 s	1.04 s	1.05 s
19	0.75 s	1.20 s	1.20 s	1.16 s	1.00 s
20	1.43 m	1.58 m	1.60 m	1.52 m	1.52 m
21	0.95 d(5.7)	1.07 d(6.6)	1.08 d(6.6)	1.00 d(6.0)	1.00 d(6.5)
22	1.38 m	1.95 m, 2.20 m	2.06 d(7.9) 2.30 dd(3.5, 16.6)	1.01 m, 1.61 m	1.01 m, 1.60 m
23	1.35 m			1.57 m, 1.23 m	1.58 m, 1.23 m
24	1.23 m			3.21 m	3.21 m
25	1.44 m	2.50 m		1.62 m	1.62 m
26	0.84 d(6.6)	1.14 d(6.9)	1.49 s	0.89 d(7.0)	0.89 d(6.5)
27	0.85 d(6.6)	1.14 d(6.9)	1.49 s	0.90 d(7.0)	0.90 d(7.5)

Table 15-2-10: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-23~15-2-26**.

H	15-2-23	15-2-24	15-2-25	15-2-26
1	1.70 dt(13.0, 3.3) 1.26 m	1.73 dt(13.0, 3.3) 1.26 m	1.92 dt(13.0, 3.5) 1.16 m	
2	1.59 m, 1.80 qd(12.5, 2.5)	1.40 m, 1.81 m	1.98 m, 2.40 qd(12.5, 2.0)	1.50, 1.75 m, 1.42 m ^①
3	3.48 ddd(11.5, 4.0, 3.0)	3.53 m	3.96 m	3.95 m

Table 15-2-10 (continued)

H	15-2-23	15-2-24	15-2-25	15-2-26
4	4.23 br s	2.23 dt(12.5, 2.5) 1.17 m	5.25 br s	2.14 dd(13.0, 12.0) 1.66 dd(5.0, 4.5)
5	1.20 dd(10.5, 3.0)	1.30 m	1.47 dd(11.0, 2.5)	
6	3.89 td(11.0, 5.3)	3.36 m	5.06 td(10.8, 3.8)	3.22 m
7	4.46 dd(13.5, 5.0) 1.56 m	4.37 dd(13.5, 5.0) 1.54 m	3.16 dd(12.3, 4.3) 1.86 m	5.36 bd(5.0)
9	1.92 dd(10.0, 8.0)	1.95 dd(10.0, 7.5)	1.03 dd(12.5, 2.3)	2.06 m
11	1.59 m, 1.55 m	1.68 m, 1.57 m	2.13 m, 1.62 m	1.55 m
12	2.14 dt(12.5, 3.5) 1.29 m	2.15 dt(13.0, 3.5) 1.31 m	2.10 m 1.23 m	2.05 m
14			1.13 d(5.5)	1.94 m
15			4.75 br t(6.0)	1.54 m
16	2.34 dd(18.5, 8.0) 2.10 dd(18.5, 12.0)	2.36 dd(19.0, 7.5) 2.12 dd(19.0, 12.3)	2.44 dt(14.5, 8.0) 1.68 m	1.28 m
17	1.49 m	1.51 m	1.06 m	1.33 m
18	0.99 s	1.00 s	1.65 s	0.62 s
19	0.95 s	0.72 s	1.86 s	1.0 s
20	1.61 m	1.62 m	1.66 s	2.0 m
21	1.03 d(6.5)	1.04 d(7.0)	1.03 d(6.5)	1.03 d(6.0)
22	1.42 m, 1.10 m	1.65 m, 1.05 m	1.48 m, 1.12 m	5.22 dd(15.0, 8.5)
23	1.44, 1.20	1.55 m, 1.24 m	1.59 m, 1.31 m	5.31 dd(15.0, 8.5)
24	1.41 m, 1.02 m	3.22 m	1.69 m, 1.21 m	1.54 m
25	1.57 m	1.62 m	1.86 m	1.60 m
26	3.40 dd(10.5, 6.0) 3.32 m	0.89 d(7.0)	3.81 dd(10.5, 5.5) 3.71 dd(10.5, 6.5)	0.87 d(7.0)
27	0.90 d(6.5)	0.90 d(7.0)	1.13 d(7.0)	0.87 d(7.0)
1'				3.61 dd(9.0, 6.5) 3.39 dd(9.0, 6.5)
2'				1.5 m
3'				1.4 m
4'				0.91 t(7.5)

① Typographic error exists in the literature, giving one more data for H-2. The placements are only to display the data.

Table 15-2-11: ¹H NMR spectroscopic data of marine cholestane-type steroids 15-2-27~15-2-31.

H	15-2-27	15-2-28	15-2-29	15-2-30	15-2-31
1	1.82 m, 1.05 m	1.62 m, 1.13 m	1.60 m, 2.08 m	7.05 d(10.0)	7.04 d(10.0)
2	1.86 m	1.84 m	2.33 m	6.22 dd(10.0, 2.0)	6.24 dd(10.0, 2.0)
	1.49 br d(6.8)	1.54 m	2.40 m		
3	3.56 m	3.62 m			

Table 15-2-11 (continued)

H	15-2-27	15-2-28	15-2-29	15-2-30	15-2-31
4	2.39 ddd(13.2, 4.8, 2.0) 2.28 m	2.41 dd(12.8, 4.8) 2.32 brt(12.8)	2.30 m 2.57 m 2.58 m	6.07 br s	6.08 br s
5					
6	5.58 dd(2.0, 2.0)	5.72 dd(5.2, 1.6)		2.35 m, 2.46 m	2.34 m, 2.45 m
7	4.13 ddd(8.8, 2.0, 2.0)	4.16 m	1.98 dd(12.9, 12.9) 2.36 m	1.10 m, 1.93 m	1.10 m, 1.93 m
8	1.63 m	1.13 m	1.83 m	1.60 m	1.59 m
9	1.16 m	1.40 m	1.32 m	1.02 m	1.04 m
11	1.53 ddd(6.4, 4.8, 2.8) 1.46 dd(12.4, 4.0)	1.38 m, 1.48 m	1.44 m, 1.65 m	1.60 m, 1.79 m	1.59 m, 1.70 m
12	1.20 m, 2.01 ddd(12.8, 3.6, 3.6)	1.95 br d(12.8) 1.19 m	α 1.24 m β 2.04 m	1.14 m, 2.01 m	1.06 m, 2.40 m
14	1.09 m	1.43 m	1.26 m	0.98 m	1.15 m
15	1.78 m, 1.35 m	1.86 m, 1.10 m	1.11 m, 1.56 m	1.17 m, 1.58 m	1.15 m, 1.64 m
16	1.81 m, 1.26 m	1.89 m, 1.27 m	1.36 m, 1.72 m	1.23 m, 1.81 m	1.38 m, 1.86 m
17	1.06 m	1.16 m	1.17 m	1.10 m	1.09 m
18	0.68 s	0.66 s	0.70 s	0.71 s	4.22 d(11.7), 3.94 d(11.7)
19	1.04 s	0.99 s	0.94 s	1.21 s	1.21 s
20	1.35 m	1.34 m	1.70 m	1.38 m	1.42 m
21	0.91 d(6.4)	0.92 d(6.4)	0.92 d(6.6)	0.88 d(6.6)	0.97 d(6.3)
22	1.31 m	1.33 m	3.60 br d(9.8)	0.99 m, 1.35 m	0.99 m, 1.35 m
23	1.38 m, 1.19 m	1.32 m, 1.19 m	1.18 m, 1.23 m	1.15 m, 1.33 m	1.14 m, 1.34 m
24	1.12 m	1.11 m	1.15 m, 1.40 m	1.37 m, 1.58 m	1.36 m, 1.58 m
25	1.47 m	1.52 m	1.24 m	2.44 m	2.45 m
26	0.86 d(6.8)	0.86 d(6.4)	0.87 d(6.6)		
27	0.86 d(6.8)	0.86 d(6.4)	0.89 d(6.6)	1.16 d(6.8)	1.17 d(7.1)
7-OOH	7.76 br s	7.68 br s			

Table 15-2-12: ^1H NMR spectroscopic data of marine cholestane-type steroids 15-2-32~15-2-35.

H	15-2-32	15-2-33	15-2-34	15-2-35
1	ax 1.28 m, eq 1.41 m	ax 1.29 m, eq 1.50 m	ax 1.21 m, eq 1.89 m	7.11 d(10.3)
2	ax 1.74 m, eq 1.59 m	1.60 m, 1.75 m	ax 1.42 m, eq 1.89 m	5.86 d(10.3)
3	4.17 p(2.5)	5.05 p(2.5), 2.06 s(OAc)	3.72 m	

Table 15-2-12 (continued)

H	15-2-32	15-2-33	15-2-34	15-2-35
4	ax 2.08 dt(2.3, 13.5) eq 1.44 m	1.37 m 1.75 m	ax 2.04 t(13.2) eq 1.48 dt(13.2, 4.0)	α 2.23 dd(17.6, 3.3) β 2.37 dd(17.6, 14.3)
5	1.63 m	1.60 m		1.91 m
6	3.62 t(2.5)	4.64 dt(4.5, 11.2) 2.02 s(OAc)	3.14 d(1.2)	1.42 m
7	3.32 d(9.0)	ax 0.95 m, eq 1.98 m	3.51 m	1.82 m
8	1.64 m	1.52 m	1.43 m	1.29 m
9	0.81 dt(2.5, 9.0)	0.80 dt(3.9, 11.0)	0.72 m	1.01 m
11	ax 1.35 m, eq 1.57 m	1.25 m, 1.53 m	ax 1.23 m, eq 1.29 m	α 1.84 m, β 1.05 m
12	ax 1.12 m, eq 2.01 m	ax 1.18 m, eq 1.98 m	ax 0.91 m, eq 1.87 m	α 2.14 m, β 1.39 m
14	1.21 m	1.15 m	1.04 m	1.21 m
15	1.43 m, 1.86 m	1.09 m, 1.57 m	1.36 m, 1.92 m	1.88 m
16	1.30 m, 1.88 m	1.30 m, 1.90 m	1.27 m, 1.85 m	1.56 m
17	1.08 m	1.45 m	1.03 m	2.01 m
18	0.70 s	0.66 s	0.65 s	α 3.55 d(9.0), β 3.63 d(9.0)
19	0.98 s	0.85 s	1.01 s	0.92 s
20	1.39 m	1.41 m	1.37 m	
21	0.94 d(6.8)	0.91 d(6.4)	0.92 d(6.8)	1.25 s
22	1.04 m, 1.41 m	3.63 ddd(1.2, 4.2, 8.6)	1.03 m, 1.40 m	5.45 d(15.6)
23	1.88 m, 1.99 m	1.40 m, 1.64 m	1.84 m, 2.02 m	5.57 dt(15.6, 7.2)
24	5.09 t(6.5)	1.41 m	5.08 t(7.0)	1.93 m
25				1.65 m
26	1.60 s	1.22 s	1.60 s	0.90 d(6.6)
27	1.68 d(0.8)	1.23 s	1.68 d(0.6)	0.91 d(6.6)

Table 15-2-13: ^1H NMR spectroscopic data of marine cholestane-type steroids 15-2-36–15-2-40.

H	15-2-36	15-2-37	15-2-38	15-2-39	15-2-40
1	1.86 m, 1.46 m	ax 1.48 m, eq 1.89 m	7.05 d(10.0)	7.05 d(10.0)	7.04 d(10.3)
2	1.93 m, 1.45 m	ax 1.49 m, eq 1.94 m	6.22 dd(10.0, 1.9)	6.22 dd(10.0, 2.0)	6.23 dd(10.3, 1.8)
3	3.58 m	3.61 m			
4	2.27 m, 1.42 m	ax 1.45 m, eq 2.31 m	6.06 brt(1.5)	6.07 brt(1.5)	6.07 brt(1.4)
5	1.75 m	1.78 m			
6	4.25 dd(9.8, 2.0)	4.27 dd(1.9, 9.8)	α 2.36 ddd(13.2, 4.2, 2.6) β 2.46 m	α 2.35 ddd(13.0, 4.4, 2.6) β 2.46 dddd(13.3, 13.3, 5.1, 1.5)	α 2.37 m β 2.47 dddd(13.3, 13.3, 5.1, 1.4)

Table 15-2-13 (continued)

H	15-2-36	15-2-37	15-2-38	15-2-39	15-2-40
7	6.51 d(2.0)	6.52 d(2.1)	α 1.04 m, β 1.84 m	α 1.02 m, β 1.94 m	α 1.04 m, β 1.94 m
8			1.60 m	1.60 m	1.62 m
9			1.04 m	1.06 m	1.04 m
11	4.16 m	4.18 m, 4.19 m	1.66 m	1.68 m	1.68 m
12	1.63 m, 1.24 m	1.27 m, 1.63 m	α 1.14 m, β 2.02 m	α 1.15 m, β 2.04 ddd(12.8, 3.6, 3.3)	α 1.18 m, β 2.02 m
14	3.23 t(9.8)	3.26 dd(8.7, 11.0)	0.98 m	1.00 m	1.00 m
15	1.57 m	1.49 m, 1.72 m	1.16 m, 1.58 m	1.15 m, 1.56 m	1.18 m, 1.62 m
16	1.80 m, 1.43 m	1.59 m	1.24 m, 1.82 m	1.26 m, 1.84 m	1.62 m, 1.80 m
17	1.63 m	1.73 m	1.08 m	1.10 m	1.14 m
18	0.66 s	0.69 s	0.73 s	0.72 s	0.75 s
19	1.10 s	1.13 s	1.22 s	1.23 s	1.23 s
20	1.39 m	2.20 m	1.38 m	1.42 m	1.88 m
21	0.96 d(6.6)	1.06 d(6.8)	0.89 d(6.4)	0.95 d(6.4)	0.97 d(6.8)
22	1.33 m, 0.98 m	5.31 m	0.98 m, 1.32 m	1.15 m, 1.52 m	4.97 dt(10.5, 3.1)
23	1.34 m, 1.13 m	5.32 m	1.16 m, 1.32 m	2.08 m, 2.20 m	2.25 br dd(14.0, 7.0) 2.37 m
24	1.11 m	1.85 m	1.38 m, 1.58 m	6.74 tq(7.7, 1.3)	6.68 br t(7.5)
25	1.49 m	1.59 m	2.42 m		
26	0.85 d(6.6)	0.87 d(6.6)			
27	0.84 d(6.6)	0.87 d(6.6)	1.14 d(6.8)	1.83 br s	1.85 br s
OMe			3.64 s	3.73 s	3.73 s
OAc	1.99 s	2.02 s			2.02 s

Table 15-2-14: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-41**–**15-2-43**.

H	15-2-41	15-2-42	15-2-43	H	15-2-41	15-2-42	15-2-43
1eq	1.68 dt(13, 3)	1.68	1.68	17	1.12	1.12	1.13
1ax	1.12	1.09	1.12	18	0.81 s	0.81 s	0.81 s
2eq	1.81	1.80	1.81	19	0.69 s	0.66 s	0.69 s
2ax	1.42	1.35	1.42	20	1.49	1.49	1.50
3ax	3.10 td (10.5, 4.8)	3.60 td (10.8, 4.4)	3.10 td (10.5, 4.7)	21	0.94 d(6.6)	0.94 d(6.6)	0.94 d(6.6)
				22	1.51, 1.20	1.52, 1.20	1.52, 1.20
4eq	1.61	1.61		23	2.17 m, 2.01 m	2.17, 2.01 m	2.17, 2.02 m
4ax	1.26	1.24	1.26	24	6.38 t(6.7)	6.38 br t(6.7)	6.40 t(6.7)
5ax	0.86	1.24	0.85	26	1.83 br s	1.83 br s	1.84 br s

Table 15-2-14 (continued)

H	15-2-41	15-2-42	15-2-43	H	15-2-41	15-2-42	15-2-43
6eq	1.69	1.33	1.69	29	0.96 d(6.3)		0.97 d(6.3)
6ax	0.98	1.20	0.98	NH	6.59 d(6.8)	6.58 d(6.8)	6.65 d(7.0)
7eq	2.38 m	2.34 m	2.39 m	30	5.53 d(6.8)	5.54 d(6.8)	5.61 d(7.0)
7ax	1.65	1.74	1.66	33	7.28 d(8.7)	7.28 d(8.7)	7.35
9ax	1.60	1.61	1.61	34	6.86 d(8.7)	6.86 d(8.7)	7.36
11eq	1.58	1.59	1.58	35			7.32
11ax	1.44	1.46	1.44	36	6.86 d(8.7)	6.86 d(8.7)	7.36
12eq	1.90 dt(12.5, 3.0)	1.91 dt(12.2, 4)	1.90 dt(12.5, 3.5)	37	7.28 d(8.7)	7.28 d(8.7)	7.35
12ax	1.08 td(13.2, 3.5)	1.09	1.09	38	3.77 s	3.77 s	
15	2.20, 2.18	2.20, 2.20	2.22, 2.18	39	3.71 s	3.72 s	3.72 s
16	1.79, 1.35	1.80, 1.33	1.80, 1.35				

Table 15-2-15: ¹H NMR spectroscopic data of marine cholestane-type steroids 15-2-44~15-2-46.

H	15-2-44	15-2-45	15-2-46	H	15-2-44	15-2-45	15-2-46
1eq	1.79	1.79	2.26 dt (13.5, 3.5)	18	0.87 s	0.88 s	2.19 br s
1ax	0.97 td(13.8, 4.3)	0.98 td(13.6, 4.3)	1.48	19	0.82 s	0.82 s	1.11 s
2eq	1.79	1.78	1.96	20	1.62	1.61	1.87
2ax	1.47	1.46	1.67 qd	21	0.92 d(6.6)	0.92 d(6.6)	0.49 d(6.6)
3ax	3.06 td (10.5, 4.7)	3.06 ddd (10.8, 10.1, 4.6)	3.16 td (10.5, 4.3)	22	1.51, 1.17	1.50, 1.15	1.38, 1.23
4ax	1.29	1.30	1.41	23	2.18 m, 2.03 m	2.18 m, 2.05 m	1.56, 1.33
5ax	0.69 td(11.4, 2.9)	0.68 td(11.5, 2.6)	1.20 td	24	6.38 t(6.7)	6.40 t(7.6)	2.65
6eq	1.72 dq (12.7, 3.0)	1.71 dq (12.6, 2.9)	1.98	26	1.84 br s	1.85 br s	5.57 br s 5.25 br s
6ax	1.08 qd (12.8, 2.1)	1.07 qd (12.6, 2.7)	1.48	28			1.08 d(7.0)
7eq	1.94	1.93	2.64	29	0.95 d(6.3)	0.95 d(6.3)	1.05 d(6.3)
7ax	1.15	1.12	2.57	NH	6.59 d(6.8)	6.65 d(7.0)	6.59 d(6.8)
8	1.97	1.97		30	5.54 d(6.8)	5.61 d(7.0)	5.54 d(6.8)
9ax	0.64 td(12.0, 2.8)	0.64 td(11.4, 2.6)		33	7.29 d(8.6)	7.37	7.28 d(8.7)
11eq	1.57	1.58	6.85 br s	34	6.86 d(8.6)	7.35	6.84 d(8.7)
11ax	1.30	1.28		35		7.33	
12eq	1.97 dt(12.7, 3.2)	1.97 dt(12.5, 3.2)		36	6.86 d(8.6)	7.35	6.84 d(8.7)
12ax	1.20	1.21		37	7.29 d(8.6)	7.37	7.28 d(8.7)

Table 15-2-15 (continued)

H	15-2-44	15-2-45	15-2-46	H	15-2-44	15-2-45	15-2-46
15	5.12 br s	5.12 br s	2.67, 2.54	38	3.77 s		3.75 s
16	2.25 ddd (15.3, 7.7, 2.9) 1.87	2.25 ddd (15.2, 7.4, 2.3) 1.89	1.88	39	3.71 s	3.72 s	3.72 s
17	1.50	1.51	3.22 dt				

Table 15-2-16: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-47** and **15-2-48**.

H	15-2-47	15-2-48	H	15-2-47	15-2-48	H	15-2-47	15-2-48
1ax	1.12	0.98	11ax	1.35	1.20	23	2.54, 2.54	2.66, 2.46
1eq	1.86	1.70	11eq	1.54	1.44	25	2.89	2.82
2ax	1.56	1.44	12ax	1.13	1.05	26	1.16 d(6.9)	3.78
2eq	1.96	1.83	12eq	1.98	1.88	27	2.85, 2.62	0.95 d(7.1)
3	3.13	3.04	14	1.43	1.33	28	3.14, 2.97	
4ax	1.43	1.31	15	1.78, 1.14	1.67, 0.98	29	3.96	
4eq	1.61	1.49	16	1.89, 1.35	1.77, 1.24	1'	3.15	2.99
5	1.74	1.62	17	1.14	1.06	2'	2.11	1.93
6ax	1.53	1.37	18	0.70 s	0.61 s	3'	3.15	2.99
6eq	1.45	1.28	19	0.86 s	0.74 s	4'	3.08	2.92
7	3.80	3.61	20	1.43	1.32	5'	1.80	1.62
8	1.44	1.29	21	0.94 d(6.9)	0.87 d(6.2)	6'	1.75	1.58
9	1.23	1.17	22	1.75, 1.23	1.62, 1.16	7'	2.98	2.81

Table 15-2-17: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-49** and **15-2-50**.

H	15-2-49	15-2-50	H	15-2-49	15-2-50	H	15-2-49	15-2-50
1ax	1.14	1.18	11ax	1.34	1.58	23	1.50, 1.44	1.71, 1.51
1eq	1.86	1.83	11eq	1.54	1.64	24	3.44	4.14
2ax	1.58	1.56	12ax	1.18		25	1.85	2.08
2eq	2.00	2.04	12eq	2.00	3.95 br s	26	4.12, 3.96	0.96 d(6.9)
3	3.14	3.16	14	1.43	1.96	27	0.99 d(6.8)	0.93 d(6.9)
4ax	1.43	1.42	15	1.77, 1.12	1.76, 1.11	1'	3.16	3.19
4eq	1.65	1.65	16	1.93, 1.32	1.90, 1.31	2'	2.11	2.16
5	1.76	1.75	17	1.18	1.82	3'	3.16	3.16
6ax	1.54	1.54	18	0.71 s	0.72 s	4'	3.09	3.08
6eq	1.46	1.46	19	0.87 s	0.85 s	5'	1.80	1.82
7	3.80	3.81	20	1.44	1.42	6'	1.76	1.75
8	1.40	1.47	21	0.97 d(6.4)	1.02 s	7'	2.99	2.99
9	1.25	1.59	22		1.49, 1.31			

Table 15-2-18: ^1H NMR spectroscopic data of marine cholestane-type steroids **15-2-51~15-2-53**.

H	15-2-51	15-2-52	15-2-53	H	15-2-51	15-2-52	15-2-53
1ax	1.15	1.12	1.14	6eq	1.46	1.45	1.46
1eq	1.85	1.86	1.86	7	3.79	3.80	3.80
2ax	1.60	1.56	1.59	8	1.44	1.41	1.40
2eq	2.03	1.96	2.01	9	1.29	1.23	1.27
3	3.11	3.12	3.12	11ax	1.37	1.34	1.36
4ax	1.44	1.42	1.44	11eq	1.56	1.54	1.55
4eq	1.64	1.63	1.65	12ax	1.18	1.14	1.16
5	1.75	1.74	1.75	12eq	2.00	1.99	2.00
6ax	1.54	1.53	1.55	14	1.44	1.47	1.44
15	1.77, 1.11	1.76, 1.11	1.76, 1.12	27	0.94 d(6.8)	1.83 dd(1.3, 1.0)	0.94 d(7.7)
16	1.92, 1.34	1.89, 1.34	1.92, 1.34	1'	3.17	3.14	3.17
17	1.18	1.16	1.17	2'	2.13	2.10	2.12
18	0.71 s	0.70 s	0.70 s	3'	3.17	3.14	3.17
19	0.87 s	0.86 s	0.86 s	4'	3.09	3.07	3.09
20	1.44	1.45	1.44	5'	1.80	1.80	1.81
21	0.97 d(6.5)	0.96 d(6.6)	0.96 d(6.3)	6'	1.77	1.76	1.81
22	1.54, 1.31	1.77, 1.28	1.50, 1.22	7'	2.98	2.98	3.09
23	1.71, 1.53	2.75, 2.67	1.71, 1.53	8'			3.14
24	4.13		4.12	9'			2.09
25	2.06		2.05	10'			3.06
26	0.96 d(6.8)	6.05, 5.84	0.95 d(7.7)				

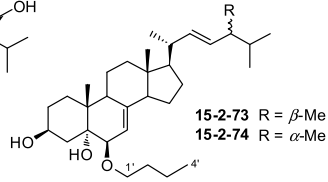
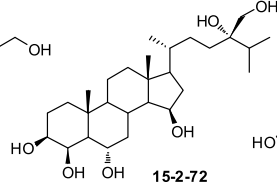
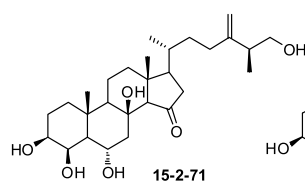
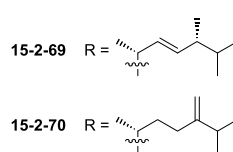
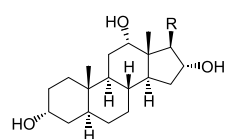
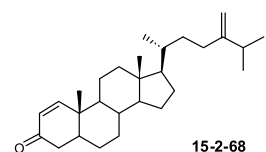
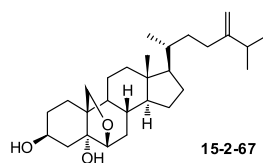
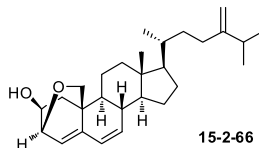
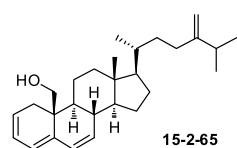
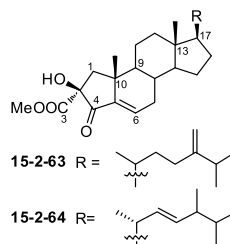
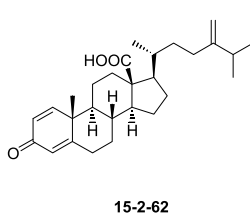
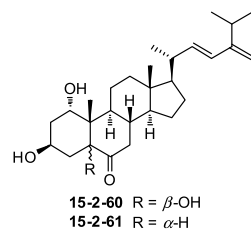
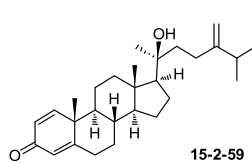
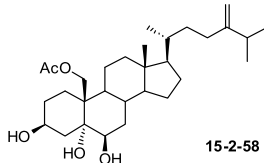
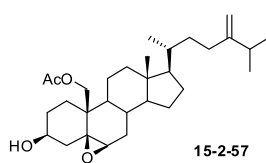
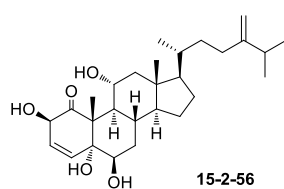
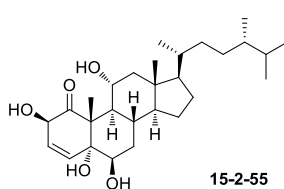
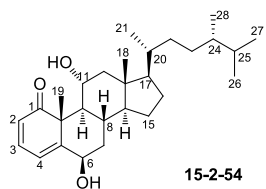
15.2.3 Marine ergostane-type steroids

Table 15-2-19: Compounds, MFs, and test solvents of marine ergostane-type steroids **15-2-54~15-2-94**.

No.	Compounds	MFs	Test solvents	References
15-2-54	stoloniferone E	$\text{C}_{28}\text{H}_{44}\text{O}_3$	CDCl_3	[58]
15-2-55	stoloniferone F	$\text{C}_{28}\text{H}_{46}\text{O}_5$	CDCl_3	[58]
15-2-56	stoloniferone G	$\text{C}_{28}\text{H}_{44}\text{O}_5$	CDCl_3	[58]
15-2-57	armatinol A	$\text{C}_{30}\text{H}_{48}\text{O}_4$	CDCl_3	[59]
15-2-58	armatinol B	$\text{C}_{30}\text{H}_{50}\text{O}_5$	CDCl_3	[59]
15-2-59	(20S)-20-hydroxyergosta-1,4,24(28)-trien-3-one	$\text{C}_{28}\text{H}_{42}\text{O}_2$	CDCl_3	[55]
15-2-60	gibberoketosterol B	$\text{C}_{28}\text{H}_{44}\text{O}_4$	CDCl_3	[60]
15-2-61	gibberoketosterol C	$\text{C}_{28}\text{H}_{44}\text{O}_3$	CDCl_3	[60]
15-2-62	norselic acid C	$\text{C}_{28}\text{H}_{40}\text{O}_3$	CDCl_3	[61]
15-2-63	anthosterone B	$\text{C}_{29}\text{H}_{44}\text{O}_4$	CDCl_3	[62]
15-2-64	(-)-phorbasterone A	$\text{C}_{29}\text{H}_{44}\text{O}_4$	CDCl_3	[62]

Table 15-2-19 (continued)

No.	Compounds	MFs	Test solvents	References
15-2-65	24-methylencholesta-2,4,6-trien-19-ol	C ₂₈ H ₄₂ O	CDCl ₃	[63]
15-2-66	24-methylencholesta-4,6-dien-3 β ,19-epoxy-2 β -ol	C ₂₈ H ₄₂ O ₂	CDCl ₃	[63]
15-2-67	24-methylencholesta-6 β ,19-epoxy-3 β ,5 α -diol	C ₂₈ H ₄₆ O ₃	CD ₃ OD	[63]
15-2-68	dendronesterone B	C ₂₈ H ₄₄ O	CDCl ₃	[64]
15-2-69	3 α ,12 α ,16 α -trihydroxy-24 <i>R</i> -methylcholest-22 <i>E</i> -ene	C ₂₈ H ₄₈ O ₃	CDCl ₃	[45]
15-2-70	3 α ,12 α ,16 α -trihydroxy-24-methylcholest-24(28)-ene	C ₂₈ H ₄₈ O ₃	CDCl ₃	[45]
15-2-71	certonardosterol Q ₁	C ₂₈ H ₄₆ O ₆	CD ₃ OD	[47]
15-2-72	certonardosterol B ₄	C ₂₈ H ₅₀ O ₆	CD ₃ OD	[47]
15-2-73	homaxisterol A ₂	C ₃₂ H ₅₄ O ₃	CD ₃ OD	[48]
15-2-74	homaxisterol A ₃	C ₃₂ H ₅₄ O ₃	CD ₃ OD	[48]
15-2-75	topsenterol B ₄	C ₂₈ H ₄₄ O ₃	CD ₃ OD	[65]
15-2-76	topsenterol B ₅	C ₂₈ H ₄₄ O ₃	CD ₃ OD	[65]
15-2-77	topsenterol C ₃	C ₂₉ H ₄₈ O ₄	CD ₃ OD	[65]
15-2-78	topsenterol D ₃	C ₂₈ H ₄₂ O ₂	CD ₃ OD	[65]
15-2-79	topsenterol E ₁	C ₂₈ H ₄₂ O ₂	CD ₃ OD	[65]
15-2-80	certonardosterol D ₂	C ₂₈ H ₄₈ O ₄	CD ₃ OD	[66]
15-2-81	certonardosterol E ₃	C ₂₈ H ₄₈ O ₄	CD ₃ OD	[66]
15-2-82	certonardosterol D ₄	C ₂₈ H ₄₈ O ₄	CD ₃ OD	[66]
15-2-83	certonardosterol C ₂	C ₂₈ H ₄₈ O ₅	CD ₃ OD	[66]
15-2-84	certonardosterol A ₂	C ₂₈ H ₄₈ O ₆	CD ₃ OD	[66]
15-2-85	24 β -methylcholasta-1,8,14,22,25-penten-3-one-5 α -ol	C ₂₈ H ₃₈ O ₂	CDCl ₃	[67]
15-2-86	29-demethylgeodisterol-3- <i>O</i> -sulfite	C ₂₇ H ₃₉ O ₆ SNa	CD ₃ OD	[68]
15-2-87	(22 <i>E</i>)-11-acetoxy-3 β ,6 α -dihydroxy-24 ξ -methyl-27- <i>nor</i> -9,11- <i>seco</i> -5 α -cholesta-7,22-dien-9-one	C ₂₉ H ₄₆ O ₅	CDCl ₃	[69]
15-2-88	11-acetoxy-3 β ,6 α -dihydroxy-24-methyl-9,11- <i>seco</i> -5 α -cholesta-7,24(28)-dien-9-one	C ₃₀ H ₄₈ O ₅	CDCl ₃	[69]
15-2-89	(22 <i>Z</i>)-11-acetoxy-3 β ,6 α -dihydroxy-24 ξ -methyl-9,11- <i>seco</i> -5 α -cholesta-7,22-dien-9-one	C ₃₀ H ₄₈ O ₅	CDCl ₃	[69]
15-2-90	polymastiamide A methyl ester	C ₃₉ H ₅₇ NO ₅	CDCl ₃	[56]
15-2-91	polymastiamide B methyl ester	C ₃₈ H ₅₅ NO ₅	CDCl ₃	[56]
15-2-92	polymastiamide E methyl ester	C ₃₈ H ₅₅ NO ₄	CDCl ₃	[56]
15-2-93	—	C ₃₉ H ₅₇ NO ₅	CDCl ₃	[56]
15-2-94	—	C ₃₈ H ₅₅ NO ₅	CDCl ₃	[56]



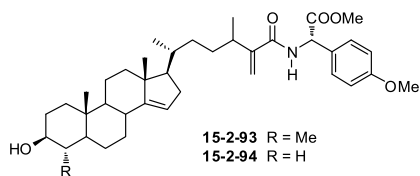
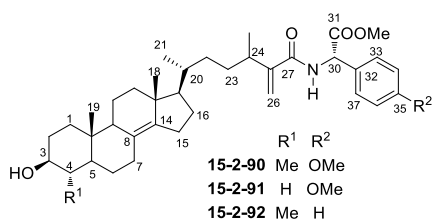
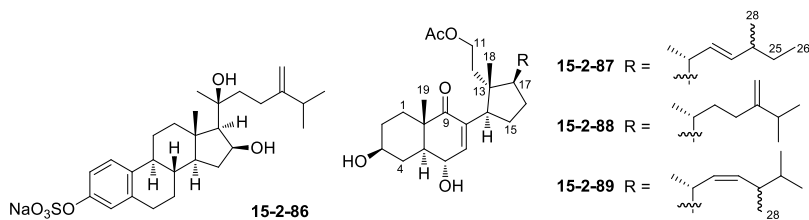
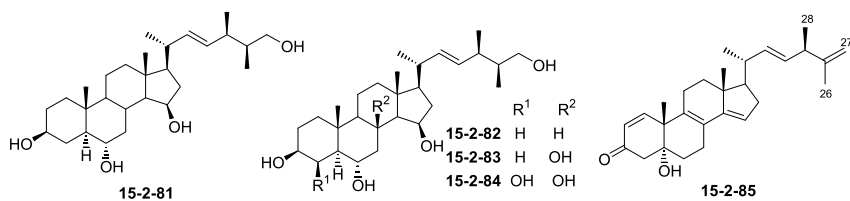
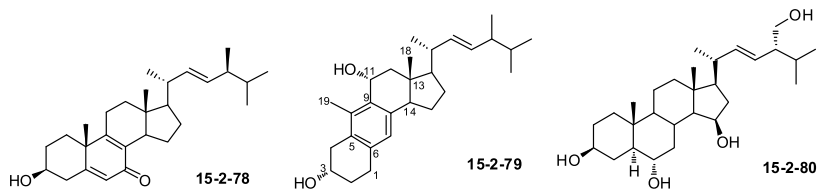
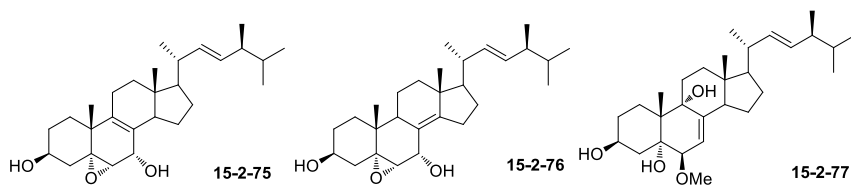


Table 15-2-20: ¹H NMR spectroscopic data of marine ergostane-type steroids **15-2-54~15-2-56**.

H	15-2-54	15-2-55	15-2-56
2	6.18 d(9.6)	7.02 dd(8.1, 1.2)	7.01 dd(8.0, 1.5)
3	6.99 dd(9.6, 6.0)	6.70 dd(8.1, 6.3)	6.70 dd(8.0, 6.5)
4	6.19 d(6.0)	4.63 dd(6.3, 1.2)	4.62 dd(6.5, 1.5)
6	4.58 br s	4.08 m	4.07 m
7	1.28 m, 2.01 m	1.69 m	—
8	2.08 m	1.85 m	—
9	1.40 m	2.00 t(9.9)	2.00 t(10.5)
11	4.04 m	4.10 m	4.10 m
12	2.39 dd(9.6, 6.0), 1.23 m	2.37 dd(12.6, 4.8), 1.31 m	2.37 dd(12.0, 4.5), 1.30 m
14	1.12 m	1.29 m	—
15	1.13 m, 1.57 m	1.12 m, 1.63 m	—
16	1.28 m, 1.87 m	1.32 m, 1.88 m	—
17	1.13 m	1.24 m	—
18	0.79 s	0.75 s	0.75 s
19	1.71 s	1.42 s	1.41 s
20	1.38 m	1.37 m	—
21	0.96 d(6.3)	0.97 d(6.6)	0.98 d(6.6)
22	0.95 m, 1.40 m	0.96 m, 1.40 m	—
23	1.39 m	0.97 m, 1.38 m	—
24	1.20 m	1.22 m	—
25	1.55 m	1.57 m	—
26	0.86 d(6.6)	0.86 d(6.9)	1.03 d(6.5)
27	0.78 d(6.6)	0.79 d(6.7)	1.04 d(6.5)
28	0.79 d(6.3)	0.80 d(6.7)	4.66 br s, 4.72 br s

Table 15-2-21: ¹H NMR spectroscopic data of marine ergostane-type steroids **15-2-57~15-2-59**.

H	15-2-57	15-2-58	15-2-59
1	α 1.41 m ^① , β 2.08 m	α 1.49 m, β 2.05 m	7.05 d(10.0)
2	α 1.39 m ^① , β 1.88 m		6.23 dd(10.0, 1.8)
3	3.79 m	4.10 m	
4	α 1.52 m, β 2.26 m	α 2.17 m, β 1.70 m	6.07 bt(1.3)
6	2.98 br s	3.54 br s	α 2.36 ddd(13.3, 4.3, 2.6) β 2.47 dddd(13.3, 13.3, 5.1, 1.3)
7	α 1.32 m, β 2.17 m		α 1.04 m, β 1.96 m
8	1.59 m	1.89 m	1.62 m
9	0.72 m	1.43 m	1.04 m
11	1.49 m, 1.55 m	1.51 m	1.68 m
12	α 1.04 m β 2.02 m	α 1.15 m ^② β 2.02 m ^②	α 1.20 m β 2.13 ddd(12.8, 3.6, 3.3)
14	0.86 m	1.12 m	1.00 m
15	1.63 m		1.64 m, 1.20 m
16	1.30 m	1.30 m	1.77 m, 1.68 m

Table 15-2-21 (continued)

H	15-2-57	15-2-58	15-2-59
17		1.18 m	1.47 m
18	0.67 s	0.68 s	0.93 s
19	4.08 d(11.4), 4.46 d(11.4)	4.49 d(12.6), 4.61 d(12.6)	1.23 s
20	1.43 m	1.45 m	
21	0.93 d(6.3)	0.94 d(6.3)	1.30 s
22	1.17 m, 1.57 m	1.19 m, 1.60 m	1.54 m
23	1.45 m		2.01 m
25	2.24 m	2.18 m	2.23 s(6.9)
26	1.02 d(6.9)	1.02 d(6.9)	1.03 d(6.9)
27	1.03 d(6.3)	1.03 d(6.3)	1.03 d(6.9)
28	4.66 s, 4.72 s	4.66 s, 4.72 s	4.74 br s, 4.67 d(1.3)
OA _c	2.10 s	2.07 s	

① Typographic errors exist in the literature, printing both data as H-2 α . Here, the placements are only to display the data.

② Typographic errors exist in the literature, printing both data as H-12 β . Here, the placements are only to display the data.

Table 15-2-22: ^1H NMR spectroscopic data of marine ergostane-type steroids 15-2-60~15-2-62.

H	15-2-60	15-2-61	15-2-62
1	4.26 dd(13.0, 3.5)	3.90 br s	7.03 d(11.0)
2	α 1.97 ddd(13.0, 13.0, 3.5) β 2.09 m	α 1.97 br d(12.0) β 1.66 ddd(12.0, 12.0, 2.5)	6.25 d(10.3)
3	4.20 br s	4.01 m	
4	α 2.34 dd(14.5, 4.0) β 1.67 br d(14.5)	α 1.51 m β 1.94 m	6.09 s
5		2.73 dd(12.5, 2.5)	
6			2.48 m, 2.37 m
7	α 2.24 dd(14.2, 14.0) β 2.42 dd(14.2, 4.7)	α 1.96 dd(13.0, 12.0) β 2.31 dd(13.0, 3.5)	1.21 m 2.76 m
8	1.79 ddd(12.0, 12.0, 4.7)	1.76 m	1.74 m
9	1.92 dd(11.5, 4.0)	1.75 m	1.11 m
11	α 2.24 m β 1.47 m	α 1.62 m β 1.39 ddd(15.0, 13.0, 2.0)	2.00 m 1.68 m
12	α 1.23 m β 2.01 dd(12.5, 3.0)	α 1.30 ddd(12.5, 12.5, 3.5) β 2.03 ddd(12.5, 3.5, 3.5)	1.56 m 1.21 m
14	1.29 m	1.28 m	1.56 m
15	α 1.52 m, β 1.52 m	α 1.51 m, β 1.09 m	1.87 m, 0.88 m
16	α 1.67 m, β 1.29 m	α 1.68 m, β 1.27 m	1.74 m, 1.53 m
17	1.22 m	1.26 m	1.42 m
18	0.69 s	0.70 s	
19	0.98 s	0.77 s	1.17 s
20	2.15 m	2.13 m	1.42 m

Table 15-2-22 (continued)

H	15-2-60	15-2-61	15-2-62
21	1.05 d(6.6)	1.06 d(6.6)	1.06 d(6.3)
22	5.56 dd(15.7, 8.8)	5.57 dd(15.5, 8.5)	2.06 m, 1.11 m
23	5.94 d(15.7)	5.94 d(15.5)	2.06 m, 1.87 m
25	2.54 sept(7.0)	2.55 sept(7.0)	
26	1.06 d(7.0)	1.06 d(7.0)	1.02 d(1.5)
27	1.08 d(7.0)	1.08 d(7.0)	1.01 d(1.5)
28	4.83 s, 4.86 s	4.83 s, 4.85 s	4.64 s, 4.72 s
1-OH	4.07 br s		
5-OH	4.50 s		

Table 15-2-23: ¹H NMR spectroscopic data of marine ergostane-type steroids 15-2-63~15-2-67.

H	15-2-63	15-2-64	15-2-65	15-2-66	15-2-67
1	2.10 d(13.5)	2.08 d(13.8)	α 2.18 m	α 1.58 m	α 1.64 m
	2.18 d(13.5)	2.17 d(13.8)	β 2.54 dd(18.0, 6.3)	β 1.62 m	β 1.46 m
2			5.77 m	3.85 dt(9.0, 1.8)	α 1.83 m, β 1.38 m
3			5.92 m	4.26 dd(5.8, 1.8)	3.84 m
4			5.77 d(5.5)	6.05 d(5.8)	α 1.67 m, β 1.85 m
6	6.73 t(3.3)	6.71 t(3.6)	6.05 dd(9.6, 2.4)	6.19 dd(9.8, 2.5)	3.66 d(4.5)
7	2.38 ddd (4.0, 6.0, 21.0)	2.38 ddd (3.9, 6.0, 20.7)	5.68 br d(9.6)	5.86 br d(9.8)	α 1.47 m β 1.84 m
8	—	—	2.35 m	1.92 m	1.60 m
9	—	—	1.42 m	1.25 m	1.58 m
11	—	—	α 1.68 m, β 1.56 m	α 1.68 m, β 1.33 m	α 1.33 m, β 1.36 m
12	—	—	α 1.15 m β 2.04 m	α 1.20 m β 2.06 dt(13.0, 4.5)	α 1.20 m β 2.03 m
14	—	—	1.18 m	1.20 m	1.22 m
15	—	—	α 1.80 m, β 1.18 m	α 1.82 m, β 1.20 m	α 1.60 m, β 1.54 m
16	—	—	α 1.90 m, β 1.28 m	α 1.92 m, β 1.33 m	α 1.86 m, β 1.29 m
17	—	—	1.18 m	1.19 m	1.17 m
18	0.73 s	0.72 s	0.74 s	0.69 s	0.74 s
19	1.20 s	1.20 s	3.79 d(11.1) 3.64 d(11.1)	3.88 d(7.3) 3.28 dd(7.3, 3.5)	3.85 d(9.0) 3.78 d(9.0)
20	—	—	1.41 m	1.44 m	1.43 m
21	0.96 d(6.7)	1.00 d(6.6)	0.95 d(6.4)	0.95 d(6.5)	0.95 d(6.5)

Table 15-2-23 (continued)

H	15-2-63	15-2-64	15-2-65	15-2-66	15-2-67
22	–	5.13~5.17 m	1.58 m, 1.15 m	1.56 m, 1.16 m	1.56 m, 1.12 m
23	–	5.13~5.27 m	2.10 m, 1.90 m	2.10 m, 1.90 m	2.12 m, 1.90 m
25	–	–	2.22 m	2.23 hept d(7.0)	2.23 hept d(7.0)
26	1.04 d(6.5)	0.89 d(6.9)	1.03 d(6.6)	1.03 d(7.0)	1.03 d(7.0)
27	1.04 d(6.5)	0.82 d(6.9)	1.03 d(6.6)	1.03 d(7.0)	1.02 d(7.0)
28	–	0.80 d(6.6)	4.72 s, 4.66 s	4.72 s, 4.66 s	4.72 s, 4.65 s
OMe	3.77 s	3.76 s			
OH	3.78 br d(1.2)	3.78 br d(0.9)			

Table 15-2-24: ^1H NMR spectroscopic data of marine ergostane-type steroids **15-2-68**–**15-2-71**.

H	15-2-68	15-2-69	15-2-70	15-2-71
1	7.15 d(10.0)	1.34	1.34	1.71 dt(13.0, 3.5) 1.00 m
2	5.85 d(10.0)	1.60, 1.35	1.64, 1.38	1.82 m, 1.55 m
3		4.01 m	4.02 m	3.42 ddd(11.0, 5.0, 3.5)
4	α 2.23 dd(17.5, 6.5) β 2.37 dd(17.5, 14.8)	1.49, 1.37	1.45, 1.39	4.24 br s
5	1.90 m	1.50	1.50	0.93 dd(11.0, 2.5)
6	1.84 m	1.16	1.16	4.10 td(11.0, 4.5)
7	1.71 m	1.60	1.64	3.07 dd(13.0, 4.5)
		0.95	0.99	1.21 dd(13.0, 11.0)
8	1.42 m	1.38	1.38	
9	0.98 m	1.16	1.19	0.82 dd(13.0, 3.0)
11	α 1.71 m, β 2.28 m	1.60, 1.50	1.62	1.71 m, 1.47 m
12	α 1.17 m β 2.04 m	3.86 t(2.8)	3.90 t(2.7)	2.15 dt(13.0, 2.8) 1.44 m
14	1.07 m	1.85 m	1.87 m	2.09 s
15	1.08 m, 1.58 m	1.53 m	1.57 m	
16	1.40 m	3.95 m	3.98 m	2.44 dd(18.5, 7.5) 1.77 dd(18.5, 9.0)
17	1.14 m	1.63 m	1.57 m	1.59 m
18	0.70 s	0.72 s	0.70 s	1.04 s
19	1.00 s	0.75 s	0.75 s	1.16 s
20	1.43 m	2.15	1.57 m	1.56 m
21	0.95 d(6.5)	1.07 d(6.0)	1.00 d(5.7)	1.01 d(6.0)
22	1.42 m	5.39 m	1.82 m, 1.31 m	1.53 m, 1.21 m
23	0.95 m	5.42 m	2.21 m, 1.92 m	2.14 m, 1.98 m
24		1.91		
25	2.17 m	1.52 m	2.23 sept(7.0)	2.25 sext(7.0)
26	1.03 (7.0)	0.84 d(6.8)	1.02 d(6.6)	3.55 dd(11.0, 6.0) 3.34 m(ov)
27	1.03 (7.0)	0.84 d(6.8)	1.01 d(6.6)	1.05 d(7.0)
28	4.66 s, 4.72 s	0.92 d(6.9)	4.72 s, 4.68 s	4.78 br s, 4.77 br s

Table 15-2-25: ¹H NMR spectroscopic data of marine ergostane-type steroids **15-2-72~15-2-74**.

H	15-2-72	15-2-73	15-2-74	H	15-2-72	15-2-73	15-2-74
1	1.64 dt(13.2, 3.3) 1.02 m	1.50 m	1.50 m	16	2.44 dt(15.0, 8.0) 1.36 m	1.28 m	1.28 m
2	1.80 m, 1.56 m	1.75 m, 1.42 m	1.75 m, 1.42 m	17	1.09 m	1.33 m	1.33 m
3	3.43 m	3.95 m	3.95 m	18	0.93 s	0.62 s	0.62 s
4	4.21 br s	2.14 dd (13.0, 12.0) 1.66 dd (5.0, 4.5)	2.14 dd (13.0, 12.0) 1.66 dd (5.0, 4.5)	19	1.06 s	1.0 s	1.0 s
				20	1.50 m	2.0 m	2.0 m
				21	0.96 d(6.5)	1.02 d(7.0)	1.03 d(7.0)
				22	1.11 m, 1.47 m	5.21 m	5.21 m
5	0.90 m			23	1.63 m, 1.34 m	5.21 m	5.21 m
6	3.90 td(11.0, 4.5)	3.22 m	3.22 m	24		1.55 m	1.55 m
7	2.35 dt(11.5, 4.3) 0.92 m	5.36 br d (5.0)	5.36 br d (5.0)	25	1.83 m	1.60 m	1.60 m
8	1.89 m			26	0.92 d(6.5)	0.84 d(7.0)	0.84 d(7.0)
9	0.67 td(11.5, 3.5)	2.06 m	2.06 m	27	0.91 d(7.0)	0.86 d(7.0)	0.86 d(6.5)
11	1.42 m 1.32 m	1.55 m	1.55 m	28	3.50 d(11.3)	0.93 d(7.0)	0.93 d(7.0)
12	1.94 dt(11.0, 2.5) 1.10 m	2.05 m	2.05 m	1'		3.61 dd(9.0, 6.5) 3.39 dd(9.0, 6.5)	3.61 dd(9.0, 6.5) 3.39 dd(9.0, 6.5)
14	0.88 m	1.94 m	1.94 m	2'		1.5 m	1.5 m
15	4.16 td(6.8, 1.5)	1.54 m	1.54 m	3'		1.4 m	1.4 m
				4'		0.91 t(7.5)	0.91 t(7.5)

Table 15-2-26: ¹H NMR spectroscopic data of marine ergostane-type steroids **15-2-75~15-2-77**.

H	15-2-75	15-2-76	15-2-77
1	1.57 m, 1.75 m	1.40 dd(13.0, 3.5) 1.68 ddd(13.0, 3.5, 3.5)	1.34 m, 2.17 dt(13.5, 4.0)
2	1.60 m, 1.90 m	1.55 m, 1.88 m	1.48 m, 1.84 m
3	3.78 m	3.74 m	3.97 m
4	1.35 m, 2.15 m	1.32 dd(13.0, 7.5), 2.10 m	1.65 ddd(13.5, 5.0, 2.0) 2.10 dd(13.5, 12.0)
6	3.19 d(2.0)	3.04 d(2.5)	3.22 dd(5.0, 2.0)
7	4.24 d(2.0)	4.40 d(2.5), 2.42 m	5.47 dd(5.0, 2.0)
11	2.06 m	1.45 m	1.58 m

Table 15-2-26 (continued)

H	15-2-75	15-2-76	15-2-77
12	1.38 m, 1.98 dd(12.0, 5.5)	1.20 m 1.95 ddd(13.5, 3.5, 3.5)	1.55 m, 1.80 m
14	2.18 m		2.50 m
15	1.28 m, 2.15 m	2.25 m, 2.66 ddd(15.5, 8.5, 8.0)	1.49 m, 1.52 m
16	1.35 m, 1.78 m	1.45 m, 1.74 m	
17	1.19 ddd(19.0, 9.0, 9.0)	1.25 m	1.35 m
18	0.68 s	0.90 s	0.63 s
19	1.13 s	0.87 s	1.02 s
20	2.04 m	2.10 m	2.05 m
21	1.02 d(6.5)	1.02 d(6.5)	1.02 d(6.5)
22	5.18 m	5.21 m	5.18 m
23	5.20 m	5.22 m	5.20 m
24	1.82 m	1.82 m	1.85 m
25	1.45 m	1.45 m	1.45 m
26	0.83 d(7.5)	0.84 d(7.0)	0.84 d(7.5)
27	0.85 d(7.5)	0.86 d(7.0)	0.86 d(7.0)
28	0.92 d(7.0)	0.93 d(7.0)	0.93 d(7.0)
OMe			3.38 s

Table 15-2-27: ^1H NMR spectroscopic data of marine ergostane-type steroids **15-2-78~15-2-80**.

H	15-2-78	15-2-79	15-2-80
1	1.25 m, 2.15 m	2.75 m, 2.86 dt(17.0, 6.0)	1.71 dt(13.0, 3.3), 1.03 m
2	1.70 ddd(25.0, 14.0, 4.0) 1.92 m	1.68 m, 1.98 m	1.75 m, 1.42 m
3	3.54 m	4.05 m	3.47 m
4	2.56 m	2.47 dd(16.5, 8.0) 3.00 dd(16.5, 4.5)	1.17 m, 2.18 m
5			1.01 m
6	6.00 br s		3.38 td(10.8, 4.5)
7		6.60 s	2.27 m, 0.91 m
8			1.88 m
9			0.72 td(11.5, 3.5)
11	2.50 m	5.09 dd(9.0, 6.5)	1.53 m, 1.33 m
12	1.51 m, 2.12 m	1.75 dd(13.5, 6.5) 2.63 dd(13.5, 9.0)	1.15 m, 1.94 m
14	2.26 m	2.92 dd(11.0, 8.0)	0.92 m
15	1.39 m, 2.53 m	1.50 m, 2.00 m	4.12 td(6.5, 2.0)
16	1.36 m, 1.82 m	1.87 m	2.24 m, 1.34 m
17	1.26 m	1.48 m	1.12 m
18	0.68 s	0.44 s	0.95 s
19	1.38 s	2.26 s	0.86 s
20	2.06 m	2.05 m	2.18 m
21	1.06 d(6.5)	1.09 d(6.5)	1.04 d(6.5)

Table 15-2-27 (continued)

H	15-2-78	15-2-79	15-2-80
22	5.20 m	5.22 m	5.30 dd(15.5, 8.5)
23	5.22 m	5.23 m	5.19 dd(15.5, 9.5)
24	1.84 m	1.85 m	1.92 m
28	0.93 d(6.5)	0.94 d(7.0)	3.54 dd(10.8, 6.3) 3.48 dd(10.8, 6.8)
25	1.45 m	1.48 m	1.79 m
26	0.84 t(6.5)	0.85 d(7.0)	0.83 d(7.0)
27	0.86 t(7.0)	0.87 d(7.0)	0.90 d(6.5)

Table 15-2-28: ¹H NMR spectroscopic data of marine ergostane-type steroids 15-2-81~15-2-84.

H	15-2-81	15-2-82	15-2-83	15-2-84
1	1.64 dt(13.0, 3.5) 0.97 m	1.71 dt(12.5, 3.3) 1.01 m	0.96 m, 1.71 m	1.71 dt(12.5, 3.5) 0.99 m
2	1.74 m, 1.42 m	1.75 m, 1.41 m	1.73 m, 1.47 m	1.83 m, 1.55 m
3	3.54 m	3.47 m	3.48 m	3.43 m
4	1.54 m, 1.74 m	1.17 m, 2.18 m	1.19 m, 2.18 dt(12.5, 3.0)	4.25 br s
5	1.13 dt(13.0, 2.5)	1.01 m	1.02 m	0.94 m
6	3.73 q(2.8)	3.38 td(11.0, 4.5)	3.69 td(10.8, 4.0)	4.15 td(11.0, 4.3)
7	2.11 dt(14.5, 3.5) 1.30 m	2.26 dt(11.5, 4.3) 0.91 m	2.36 dd(12.0, 4.0) 1.25 m	2.43 dd(12.0, 4.5) 1.29 m
8	1.95 m	1.86 m		
9	0.72 td(11.3, 3.5)	0.72 td(11.0, 3.5)	0.83 dd(11.0, 3.0)	0.82 dd(12.0, 2.5)
11	1.55 m, 1.39 m	1.52 m, 1.31 m	1.80 qd(13.0, 3.0), 1.50 m	1.78 m, 1.43 m
12	1.94 m 1.24 m	1.93 dt(12.5, 3.3) 1.12 m	1.97 dt(12.5, 3.3) 1.17 m	1.96 dt(12.5, 3.5) 1.17 m
14	1.07 dd(11.0, 9.5)	0.92 m	1.03 m	1.01 d(6.0)
15	3.85 td(9.5, 3.0)	4.12 td(6.5, 2.5)	4.37 td(6.5, 2.0)	4.38 td(6.5, 2.0)
16	1.57 m, 1.87 m	1.31 m, 2.21 m	1.36 m, 2.18 m	1.35 ddd(15.0, 10.5, 2.5) 2.19 dt(15.0, 7.8)
17	1.45 m	1.11 m	1.02 m	1.01 m
18	0.76 s	0.95 s	1.28 s	1.27 s
19	1.03 s	0.87 s	0.98 s	1.15 s
20	2.00 m	2.13 m	2.13 m	2.13 m
21	1.00 d(6.0)	1.01 d(6.5)	0.99 d(7.0)	0.99 d(6.5)
22	5.20 dd(15.0, 8.0)	5.20 dd(15.0, 8.5)	5.18 dd(15.0, 8.3)	5.18 dd(15.0, 8.3)
23	5.25 dd(15.0, 7.0)	5.26 dd(15.0, 7.3)	5.26 dd(15.0, 8.0)	5.26 dd(15.0, 8.0)
24	2.07 m	2.08 m	2.08 sext(6.8)	2.08 m
25	1.53 m	1.52 m	1.52 m	1.52 m
26	3.56 dd(11.0, 5.8) 3.26 dd(15.0, 7.0)	3.57 dd(11.0, 5.5) 3.28 m	3.56 dd(11.0, 5.0) 3.27 dd(11.0, 7.3)	3.56 dd(10.8, 5.3) 3.27 dd(10.8, 7.5)
27	0.87 d(6.5)	0.88 d(7.0)	0.87 d(7.0)	0.87 d(7.0)
28	0.92 d(6.5)	0.93 d(7.0)	0.92 d(7.0)	0.92 d(7.0)

Table 15-2-29: ^1H NMR spectroscopic data of marine ergostane-type steroids **15-2-85~15-2-89**.

H	15-2-85	15-2-86	15-2-87	15-2-88	15-2-89
1	6.55 d(9.6)	7.24 d(8.5)	1.86 m, 1.46 m	1.88 m, 1.45 m	1.87 m, 1.46 m
2	6.42 d(9.6)	7.05 d(8.5)	1.91 m, 1.44 m	1.90 m, 1.46 m	1.91 m, 1.44 m
3			3.57 m	3.58 m	3.58 m
4	2.82 d(18.0) 2.13 d(18.0)	7.02 br s	2.28 m 1.43 m	2.28 m 1.42 m	2.28 m 1.41 m
5			1.76 m	1.76 m	1.74 m
6	1.51 m	2.87	4.24 dd(9.9, 1.7)	4.25 dd(9.9, 1.6)	4.25 dd(9.9, 1.9)
7	2.19 m, 1.36 m	α 1.49, β 1.92	6.49 d(1.7)	6.51 d(1.6)	6.50 d(1.9)
8		1.49			
9		2.23			
11	2.21 m	α 2.23, β 1.55	4.15 m	4.15 m	4.15 m
12	1.86 m, 1.40 m	α 1.44, β 2.28	1.60 m, 1.23 m	1.63 m, 1.21 m	1.62 m, 1.21 m
14		1.15	3.22 t(9.8)	3.24 t(10.0)	3.23 t(10.0)
15	5.68 br s	1.40, 2.35	1.57 m	1.59 m	1.58 m
16	2.49 m, 2.42 m	4.63	1.70 m, 1.44 m	1.85 m, 1.43 m	1.70 m, 1.42 m
17	1.74 m	1.38	1.69 m	1.67 m	1.67 m
18	0.97 s	1.19 s	0.65 s	0.66 s	0.66 s
19	1.10 s		1.09 s	1.10 s	1.10 s
20	1.99 br m		2.15 m	1.44 m	2.15 m
21	1.11 d(7.0)	1.34 s	1.01 d(6.8)	0.99 d(6.8)	1.02 d(6.8)
22	5.28 dd(7.0, 15.1)	1.82 dt(5.0, 12.3) 1.93	5.21 dd(15.1, 8.0)	1.54 m 1.13 m	5.21 m
23	5.33 dd(5.6, 15.1)	2.12	5.16 dd(15.1, 7.4)	2.09 m, 1.86 m	5.20 m
24	2.74 m		1.91 m		1.86 m
25		2.32	1.24 m	2.20 m	1.45 m
26	4.72 br s	1.08 d(7.4)	0.81 t(6.7)	1.00 d(6.7)	0.81 d(6.9)
27	1.69 s	1.08 d(7.4)		1.01 d(6.7)	0.80 d(6.9)
28	1.07 d(6.6)	4.74 br s, 4.77 br s	0.90 d(6.7)	4.70 br s, 4.63 br s	0.89 d(6.8)
OAc			1.99 s	1.99 s	1.99 s

Table 15-2-30: ^1H NMR spectroscopic data of marine ergostane-type steroids **15-2-90~15-2-94**.

H	15-2-90	15-2-91	15-2-92	15-2-93	15-2-94
1eq	1.68	1.67 dt(13.8, 3.4)	1.68	1.77	1.75
1ax	1.12	1.08	1.13	0.97 td(13.4, 4.3)	0.95 td(13.8, 4.3)
2eq	1.81 dq(13, 4)	1.80	1.81 dq(13, 4)	1.79	1.78
2ax	1.42	1.33	1.42	1.47	1.54

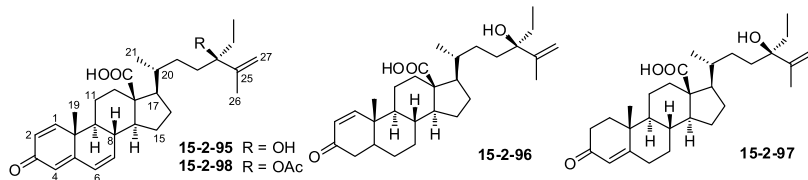
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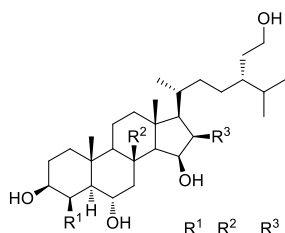
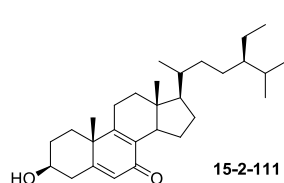
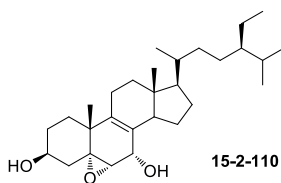
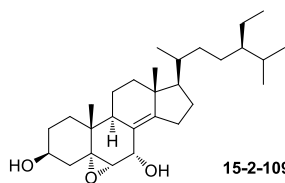
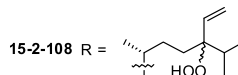
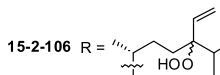
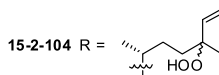
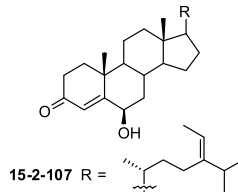
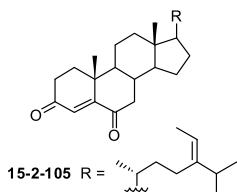
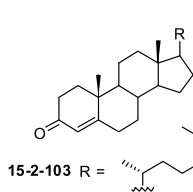
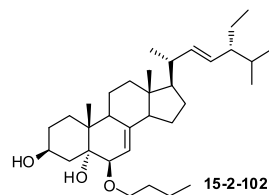
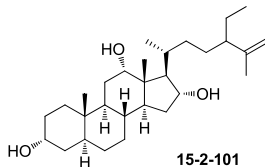
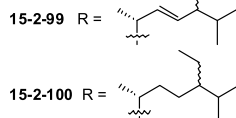
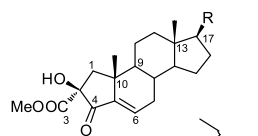
H	15-2-90	15-2-91	15-2-92	15-2-93	15-2-94
3ax	3.11 td(10.8, 4.4)	3.60 tt(10.5, 5.0)	3.11 td(10.8, 4.5)	3.06 ddd(11.1, 9.9, 4.3)	3.57 tt(10.8, 4.7)
4eq					1.57
4ax	1.26	1.25	1.26	1.28	1.27
5ax	0.84	1.24	0.84	0.68 td(11.3, 2.9)	1.07 td(11.4, 2.9)
6eq	1.69	1.33	1.69	1.71 dq(12.5, 3.3)	1.32
6ax	0.98	1.19	0.97	1.07	1.32
7eq	2.38 m	2.34 m	2.38	1.92 dq(13.8, 3.3)	1.87
7ax	1.65	1.74	1.64	1.14	1.15
8				1.93	1.97
9ax	1.61	1.61	1.61	0.63 td(11.6, 2.8)	0.64 td(12.0, 2.8)
11eq	1.58	1.58	1.58	1.55	1.53
11ax	1.43	1.45	1.43	1.28	1.29
12eq	1.88 dt(12.2, 4.0)	1.88 dt(12.7, 3.6)	1.88 dt(12.2, 4.0)	1.94 dt(12.6, 3.1)	1.94 dt(12.8, 3.2)
12ax	1.06	1.08	1.06	1.16	1.18
15	2.16, 2.16	2.17 m, 2.14 m	2.16, 2.16	5.08 br s	5.08 br s
16	1.73	1.73	1.72	2.18 ddd(15.8, 9.6, 2.2)	2.17 ddd(15.4, 7.5, 1.9)
	1.27	1.27	1.27	1.79	1.77
17	1.06	1.06	1.06	1.43	1.44
18	0.78 s	0.78 s	0.78	0.84 s	0.87 s
19	0.69 s	0.66 s	0.69	0.82 s	0.82 s
20	1.37	1.37	1.37	1.50	1.51
21	0.86 d(6.6)	0.86 d(6.6)	0.85 d(6.7)	0.84 d(6.4)	0.84 d(6.4)
22	1.36, 1.01	1.36, 1.01	1.36, 1.01	1.34 m, 0.96	1.35, 0.96
23	1.52, 1.17	1.50, 1.18	1.50, 1.16	1.53, 1.18	1.53, 1.19
24	2.57 dt(6.8)	2.57 dt(6.8)	2.57 dt(6.9, 6.9)	2.58 dt(6.6)	2.58 dt(6.7)
26	5.57 br s, 5.22 br s	5.57 br s, 5.22 br s	5.58 br s, 5.23 br s	5.57 br s, 5.23 br s	5.57 br s, 5.23 br s
28	1.05 d(6.8)	1.05 d(6.8)	1.05 d(6.8)	1.05 d(6.9)	1.05 d(6.9)
29	0.97 d(6.3)		0.97 d(6.4)	0.95 d(6.3)	
NH	6.65 d(7.1)	6.65 d(7.2)	6.72 d(7.1)	6.67 d(7.1)	6.67 d(7.1)
30	5.55 d(7.1)	5.55 d(7.2)	5.61 d(7.1)	5.54 d(7.1)	5.54 d(7.1)
33	7.27 d(8.7)	7.27 d(8.8)	7.34	7.27 d(8.8)	7.27 d(8.8)
34	6.85 d(8.7)	6.85 d(8.8)	7.35	6.85 d(8.8)	6.85 d(8.8)
35			7.31		
36	6.85 d(8.7)	6.85 d(8.8)	7.35	6.85 d(8.8)	6.85 d(8.7)
37	7.27 d(8.7)	7.27 d(8.8)	7.34	7.27 d(8.8)	7.27 d(8.7)
38	3.77 s	3.77 s		3.77 s	3.77 s
39	3.72 s	3.72 s	3.73	3.72 s	3.72 s

15.2.4 Marine stigmastane-type steroids

Table 15-2-31: Compounds, MFs, and test solvents of marine stigmastane-type steroids **15-2-95**–**15-2-117**.

No.	Compounds	MFs	Test solvents	References
15-2-95	norselic acid A	C ₂₉ H ₄₀ O ₄	CDCl ₃	[61]
15-2-96	norselic acid B	C ₂₉ H ₄₄ O ₄	CDCl ₃	[61]
15-2-97	norselic acid D	C ₂₉ H ₄₄ O ₄	CDCl ₃	[61]
15-2-98	norselic acid E	C ₃₁ H ₄₂ O ₅	CDCl ₃	[61]
15-2-99	phorbasterone C	C ₃₀ H ₄₆ O ₄	CDCl ₃	[62]
15-2-100	phorbasterone D	C ₃₀ H ₄₈ O ₄	CDCl ₃	[62]
15-2-101	3 α ,12 α ,16 α -trihydroxy-24 ξ -ethylcholest-25-ene	C ₂₉ H ₅₀ O ₃	CDCl ₃	[45]
15-2-102	homaxisterol A ₄	C ₃₃ H ₅₆ O ₃	CD ₃ OD	[48]
15-2-103	24-ethylcholesta-4,24(28)-dien-3-one	C ₂₉ H ₄₆ O	CDCl ₃	[70]
15-2-104	24 ξ -hydroperoxy-24-ethylcholesta-4,28(29)-dien-3-one	C ₂₉ H ₄₆ O ₃	CDCl ₃	[70]
15-2-105	24-ethylcholesta-4,24(28)-dien-3,6-dione	C ₂₉ H ₄₄ O ₂	CDCl ₃	[70]
15-2-106	24 ξ -hydroperoxy-24-ethylcholesta-4,24(28)-dien-3,6-dione	C ₂₉ H ₄₄ O ₄	CDCl ₃	[70]
15-2-107	6 β -hydroxy-24-ethylcholesta-4,24(28)-dien-3-one	C ₂₉ H ₄₆ O ₂	CDCl ₃	[70]
15-2-108	24 ξ -hydroperoxy-6 β -hydroxy-24-ethylcholesta-4-28(29)-dien-3-one	C ₂₉ H ₄₆ O ₄	CDCl ₃	[70]
15-2-109	5 α ,6 α -epoxy-24 <i>R</i> *-ethylcholest-8(14)-en-3 β ,7 α -diol	C ₂₉ H ₄₈ O ₃	CDCl ₃	[71]
15-2-110	5 α ,6 α -epoxy-24 <i>R</i> *-ethylcholest-8-en-3 β ,7 α -diol	C ₂₉ H ₄₈ O ₃	CDCl ₃	[71]
15-2-111	3 β -hydroxy-24 <i>R</i> *-ethylcholesta-5,8-dien-7-one	C ₂₉ H ₄₆ O ₂	CDCl ₃	[71]
15-2-112	certonardosterol D ₃	C ₂₉ H ₅₂ O ₄	CD ₃ OD	[66]
15-2-113	certonardosterol N ₁	C ₂₉ H ₅₂ O ₅	CD ₃ OD	[66]
15-2-114	certonardosterol O ₁	C ₂₉ H ₅₂ O ₆	CD ₃ OD	[66]
15-2-115	certonardosterol P ₁	C ₂₉ H ₅₂ O ₇	CD ₃ OD	[66]
15-2-116	certonardosterol E ₂	C ₂₉ H ₅₂ O ₄	CD ₃ OD	[66]
15-2-117	geodisterol-3- <i>O</i> -sulfite	C ₂₈ H ₄₁ NaO ₆ S	CD ₃ OD	[68]





15-2-112 H H H

15-2-113 H H OH

15-2-114 H OH OH

15-2-115 OH OH OH

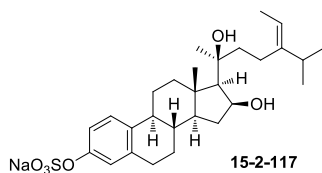
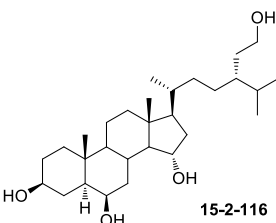


Table 15-2-32: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-95~15-2-98**.

H	15-2-95	15-2-96	15-2-97	15-2-98
1	7.03 d(10.2)	7.11 d(10.2)	1.98 m, 1.68 m	7.04 d(10.3)
2	6.25 dd(10.1, 1.8)	5.86 d(10.3)	2.35 m, 2.28 m	6.27 dd(1.5, 10.3)
4	6.01 s	2.22 m, 2.36 m	5.73 m	6.02 s
5		1.94 m		
6	6.21 dd(10.0, 2.9)	1.43 m	2.35 m	6.23 dd(2.9, 9.9)
7	6.07 dd(9.7, 1.4)	1.83 m, 1.61 m	1.91 m, 1.05 m	6.08 d(9.2)
8	2.35 m	1.54 m	1.40 m	2.35 m
9	1.49 m	1.54 m	0.95 m	1.50 m
11	1.87 m, 1.43 m	1.92 m, 1.56 m	1.40 m, 1.69 m	1.96 m
12	2.74 m, 1.31 m	2.73 m, 1.16 m	2.69 m, 1.14 m	2.77 m, 1.15 m
14	1.58 m	1.43 m	1.53 m	1.57 m
15	2.01 m, 1.46 m	1.91 m, 1.68 m	1.87 m, 1.57 m	1.96 m
16	1.81 m, 1.54 m	1.94 m, 1.68 m	1.93 m, 1.22 m	0.93 m
17	1.58 m	1.54 m	1.40 m	1.57 m
19	1.09 s	0.94 s	1.10 s	1.12 s
20	1.36 m	1.35 m	1.68 m	1.43 m
21	0.98 d(6.5)	1.02 d(6.4)	1.00 d(6.6)	1.01 d(6.8)
22	1.95 m, 1.58 m	1.54 m, 0.89 m	1.83 m, 1.60 m	1.81 m, 1.57 m
23	1.62 m, 1.31 m	1.61 m, 1.35 m	1.98 m, 1.68 m	1.96 m, 1.81 m
26	4.87 s, 4.91 s	4.90 s, 4.94 s	4.93 s, 4.89 s	4.93 s, 4.96 s
27	1.62 s	1.66 s	1.65 s	1.65 s
28	1.49 m, 1.58 m	1.61 m, 1.54 m	1.53 m, 1.60 m	1.81 m, 2.06 m
29	0.77 t(7.1)	0.81 t(7.3)	0.79 t(7.4)	0.73 t(7.6)
OAc				2.04 s

Table 15-2-33: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-99~15-2-102**.

H	15-2-99	15-2-100	15-2-101	15-2-102
1	2.08 d(14) 2.17 d(14)	2.08 d(13.6) 2.17 d(13.6)	1.34	1.50 m
2			1.61, 1.37	1.75 m, 1.42 m
3			4.01 p(2.8)	3.95 m
4			1.46, 1.35	2.14 dd(13.0, 12.0) 1.66 dd(5.0, 4.5)
5			1.50	
6	6.72 t(3.6)	6.72 t(3.6)	1.17, 1.08	3.22 m
7	2.37 ddd(3.6, 5.7, 21.0) 1.85 ddd(3.6, 9.3, 21.0)	2.38 ddd(4.0, 6.0, 21.0) 1.85 ddd(4.0, 9.0, 1.0)	1.62 m 1.00 qd(12.3, 4.1)	5.36 br d(5.0)
8	—	—	1.38	
9	—	—	1.16 td(12.3, 2.6)	2.06 m

Table 15-2-33 (continued)

H	15-2-99	15-2-100	15-2-101	15-2-102
11	—	—	1.63, 1.49	1.55 m
12	—	—	3.89 t(3.0)	2.05 m
14	—	—	1.84 ddd(12.1, 10.6, 7.2)	1.94 m
15	—	—	1.56	1.54 m
16	—	—	3.96 ddd(2.3, 5.0, 6.9)	1.28 m
17	—	—	1.53	1.33 m
18	0.72 s	0.71 s	0.68 s	0.62 s
19	1.20 s	1.18 s	0.74 s	1.0 s
20	—	—	1.49	2.0 m
21	1.02 d(6.4)	0.91 d(6.4)	0.94 d(5.8)	1.05 d(7.0)
22	5.14 ddd(2.8, 8.4, 15.2)	—	1.08	5.19 dd(15.0, 8.5)
23	5.01 ddd(2.8, 8.4, 15.2)	—	1.42, 1.24	5.06 dd(15.0, 8.5)
24	—	—	1.87 dt(9.7, 5.0)	1.55 m
25	—	—	—	1.60 m
26	0.83 d(6.6)	0.82 d(6.6)	4.72 dq(2.7, 1.5) 4.65 br d(2.7)	0.81 d(7.0)
27	0.79 d(6.6)	0.79 d(6.6)	1.56 s	0.86 d(7.0)
28	—	—	1.31	1.45 m, 1.22 m
29	—	—	0.78 t(7.4)	0.84 t(7.5)
1'				3.61 dd(9.0, 6.5)
2'				3.39 dd(9.0, 6.5)
3'				1.5 m
4'				1.4 m
OMe	3.76	3.76 s		0.91 t(7.5)
OH	3.78 br d(0.9)	3.78 br s		

Table 15-2-34: ¹H NMR spectroscopic data of marine stigmastane-type steroids 15-2-103~15-2-105.

H	15-2-103	15-2-104	15-2-105
4	5.72 s	5.73 s	6.17 s
18	0.72 s	0.72 s	0.73 s
19	1.18 s	1.18 s	1.17 s
21	0.99 d(6.6)	0.96 d(6.3)	1.00 d(6.6)
25	2.22 m	—	2.20 m
26	0.98 d(6.9)	0.86 d(6.6)	0.98 d(6.9)
27	0.98 d(6.9)	0.88 d(6.6)	0.98 d(6.9)
28	5.19 q(6.6)	5.75 dd(17.7, 11.4)	5.19 q(6.9)
29	1.58 d(6.6)	5.16 dd(17.7, 1.5)	1.57 d(6.9)
		5.28 dd(11.4, 1.5)	
OOH		7.01 s, 7.02 s	

Table 15-2-35: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-106~15-2-108**.

H	15-2-106	15-2-107	15-2-108
4	6.18 s	5.82 s	5.82 s
6		4.36 br s	4.35 br s
18	0.73 s	0.75 s	0.75 s
19	1.17 s	1.38 s	1.38 s
21	0.98 d(6.6)	0.99 d(6.3)	0.96 d(6.9)
25		2.20 m	
26	0.87 d(6.9)	0.98 d(6.6)	0.86 d(6.9)
27	0.89 d(6.9)	0.98 d(6.6)	0.89 d(6.9)
28	5.74 dd(17.7, 11.4)	5.19 q(6.6)	5.75 dd(17.4, 11.1)
29	5.16 dd(17.7, 1.5)	1.57 d(6.6)	5.16 dd(17.4, 1.5)
	5.29 dd(11.4, 1.5)		5.28 dd(11.1, 1.5)
OOH			7.01 s, 7.02 s

Table 15-2-36: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-109~15-2-111**.

H	15-2-109	15-2-110	15-2-111
1	α 1.40 m, β 1.70 m	α 1.62 m, β 1.82 m	1.38 m, 1.27 m
2	α 1.93 m, β 1.52 m	α 1.97 m, β 1.60 m	1.97 m, 1.72 m
3	3.91 m	3.95 m	3.67 m
4	α 1.41 m	α 1.44 m	2.60 m
	β 2.13 dd(12.9, 11.5)	β 2.18 m	2.54 dd(11.0, 1.4)
6	3.15 d(3.5)	3.31 d(2.5)	6.04 d(1.4)
7	4.41 m	4.23 m	
9	2.34 m		
11	1.57 m, 1.48 m	2.14 m	—
12	1.92 m, 1.15 m	1.97 m, 1.41 m	—
14		2.15 m	2.40 m
15	2.64 m, 2.34 m	2.04 m, 1.14 m	2.61 m, 1.43 m
16	1.92 m, 1.40 m	1.67 m, 1.30 m	
17	1.21 m	1.15 m	1.21 m
18	0.85 s	0.57 s	0.64 s
19	0.86 s	1.14 s	1.34 s
20	1.45 m	1.38 m	1.41 m
21	0.93 d(6.6)	0.93 d(6.6)	0.96 d(6.6)
22	1.03 m, 1.30 m	1.32 m, 0.91 m	
23	1.04 m, 1.32 m	1.34 m, 0.96 m	
24	0.92 m	0.92 m	0.92 m
25	1.67 m	1.62 m	1.69 m
26	0.80 d(6.8)	0.80 d(6.8)	0.81 d(6.9)
27	0.82 d(6.8)	0.82 d(6.8)	0.83 d(6.9)
28	1.32 m, 1.15 m	1.32 m, 1.15 m	1.32 m, 1.15 m
29	0.85 t(7.0)	0.85 t(7.0)	0.86 t(7.4)

Table 15-2-37: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-112~15-2-115**.

H	15-2-112	15-2-113	15-2-114	15-2-115
1	1.03 m, 1.71 m	1.01 m, 1.72 m	0.97 m, 1.71 dt(12.5, 3.5)	0.98 m, 1.70 dt(12.5, 2.5)
2	1.42 m, 1.75 m	1.42 m, 1.76 m	1.47 m, 1.72 m	1.55 m, 1.82 qd(13.0, 3.5)
3	3.47 m	3.47 m	3.48 m	3.42 ddd(12.0, 5.0, 3.5)
4	1.16 m, 2.18 dt(12.5, 2.5)	1.17 m, 2.19 dt(12.5, 2.0)	1.21 m, 2.19 dt(11.7, 2.3)	4.25 br s
5	1.01 m	1.01 m	1.04 m	0.94 m
6	3.38 td(10.8, 5.0)	3.39 td(10.5, 4.8)	3.71 td(11.0, 4.5)	4.18 td(11.0, 4.3)
7	0.90 m, 2.27 dt(12.5, 3.8)	2.32 dt(12.0, 4.0)	2.39 dd(12.0, 4.3)	2.46 dd(12.5, 4.0)
8	1.87 m	0.94 m 1.89 m	1.33 m	1.35 m
9	0.71 td(11.3, 3.5)	0.72 td(10.5, 3.0)	0.84 dd(11.0, 3.5)	0.82 dd(11.0, 3.0)
11	1.52 m, 1.33 m	1.48 m, 1.30 m	1.77 m, 1.47 m	1.77 m, 1.42 m
12	1.95 dt(12.5, 3.0) 1.11 m	1.05 m, 1.92 m	1.95 dt(12.5, 3.3) 1.13 m	1.93 dt(13.0, 3.5) 1.11 m
14	0.89 m	0.88 m	1.02 m	1.01 m
15	4.16 td(6.8, 2.0)	4.10 br t(6.5)	4.36 dd(7.0, 5.5)	4.37 dd(7.0, 5.5)
16	2.39 dt(14.5, 8.3), 1.35 m	4.24 br t(7.3)	4.21 t(7.0)	4.21 t(7.0)
17	1.07 m	1.03 m	0.96 m	0.95 m
18	0.93 s	0.94 s	1.24 s	1.23 s
19	0.86 s	0.86 s	0.98 s	1.15 s
20	1.49 m	1.89 m	1.90 m	1.90 m
21	0.95 d(7.0)	0.95 d(7.0)	0.95 d(6.5)	0.94 d(6.5)
22	1.41 m, 1.02 m	1.68 m, 1.10 m	1.69 m, 1.11 m	1.68 m, 1.11 m
23	1.38 m, 1.11 m	1.41 m, 1.15 m	1.41 m, 1.15 m	1.41 m, 1.16 m
24	1.18 m	1.23 m	1.22 m	1.22 m
25	1.71 m	1.76 m	1.77 m	1.76 m
26	0.84 d(7.0)	0.84 d(7.0)	0.84 d(7.0)	0.84 d(7.0)
27	0.87 d(7.0)	0.88 d(7.0)	0.88 d(7.0)	0.88 d(7.0)
28	1.58 m, 1.33 m	1.58 m, 1.34 m	1.58 m, 1.35 m	1.58 m, 1.35 m
29	3.55 m	3.55 m	3.57 m	3.57 m

Table 15-2-38: ^1H NMR spectroscopic data of marine stigmastane-type steroids **15-2-116** and **15-2-117**.

H	15-2-116	15-2-117	H	15-2-116	15-2-117
1	1.64 dt(13.5, 3.8) 0.97 m	7.23 d(8.4)	17	1.41 m	1.39 d(7.8)
2	1.73 m, 1.42 m	7.05 d(8.4)	18	0.74 s	1.19 s
3	3.53 m		19	1.03 s	
4	1.74 m 1.55 m	7.02 br s	20	1.35 m	
5	1.13 dt(13.5, 2.3)		21	0.94 d(5.5)	1.37 s
6	3.74 q(2.8)	2.87	22	1.38 m 1.03 m	1.70 dt(4.2, 13.2), 1.87
7	2.12 dt(14.0, 3.3) 1.31 m	α 1.34 β 1.91	23	1.37 m 1.08 m	2.08 dt(3.6, 12.6) 2.15 dt(5.4, 12.6)
8	1.94 m	1.48	24	1.17 m	
9	0.71 td(11.0, 3.3)	2.22	25	1.70 m	2.27
11	1.52 m, 1.37 m	α 2.29, β 1.56	26	0.84 d(7.0)	1.04 d(7.2)
12	1.96 m, 1.24 m	α 1.41, β 2.25	27	0.87 d(7.0)	1.04 d(7.2)
14	1.06 t(9.3)	1.12	28	1.57 m, 1.32 m	5.25 q(6.6)
15	3.87 td(9.3, 3.3)	1.40, 2.34	29	3.55 m	1.63 d(6.6)
16	1.87 m, 1.70 m	4.67			

15.2.5 Some additional marine steroids

Table 15-2-39: Compounds, MFs, and test solvents of marine steroids **15-2-118~15-2-175**.

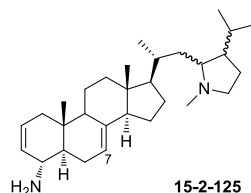
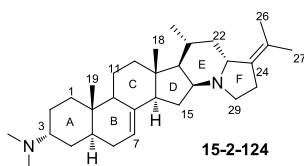
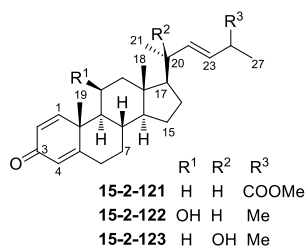
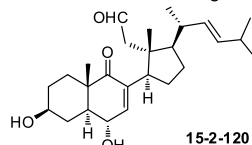
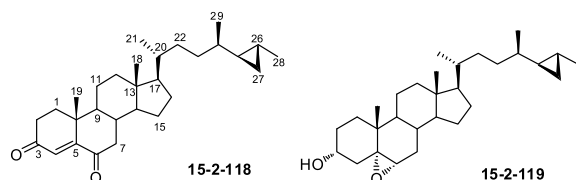
No.	Compounds	MFs	Test solvents	References
15-2-118	petrosterol-3,6-dione	$\text{C}_{29}\text{H}_{44}\text{O}_2$	CDCl_3	[72]
15-2-119	5 α ,6 α -epoxy-petrosterol	$\text{C}_{29}\text{H}_{48}\text{O}_2$	CDCl_3	[72]
15-2-120	—	$\text{C}_{26}\text{H}_{40}\text{O}_4$	CDCl_3	[52]
15-2-121	methyl (22 <i>E</i>)-3-oxo-24-norcholesta-1,4,22-trien-26-oate	$\text{C}_{27}\text{H}_{38}\text{O}_3$	CDCl_3	[55]
15-2-122	(22 <i>E</i>)-11 β -hydroxy-24-norcholesta-1,4,22-trien-3-one	$\text{C}_{26}\text{H}_{38}\text{O}_2$	CDCl_3	[55]
15-2-123	(20 <i>S</i> ,22 <i>E</i>)-20-hydroxy-24-norcholesta-1,4,22-trien-3-one	$\text{C}_{26}\text{H}_{38}\text{O}_2$	CDCl_3	[55]
15-2-124	plakinamine I	$\text{C}_{31}\text{H}_{50}\text{N}_2$	CD_3OD	[73]
15-2-125	plakinamine J	$\text{C}_{30}\text{H}_{50}\text{N}_2$	CD_3OD	[73]
15-2-126	plakinamine K	$\text{C}_{32}\text{H}_{52}\text{N}_2\text{O}_2$	CD_3OD	[73]
15-2-127	dihydroplakinamine K	$\text{C}_{32}\text{H}_{54}\text{N}_2\text{O}_2$	CD_3OD	[73]
15-2-128	demethylincisterol A ₁	$\text{C}_{20}\text{H}_{30}\text{O}_3$	CD_3OD	[48]
15-2-129	incisterol	$\text{C}_{21}\text{H}_{32}\text{O}_3$	CD_3OD	[48]
15-2-130	hippuristerone G	$\text{C}_{35}\text{H}_{54}\text{O}_9$	CDCl_3	[74]
15-2-131	hippuristerone H	$\text{C}_{31}\text{H}_{50}\text{O}_6$	CDCl_3	[74]

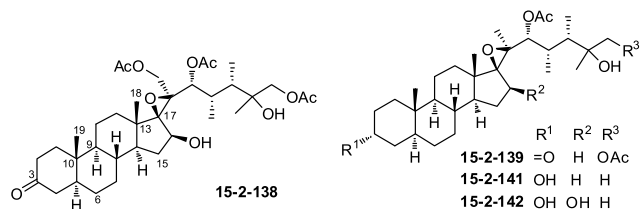
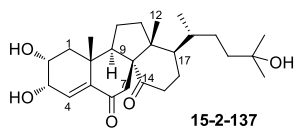
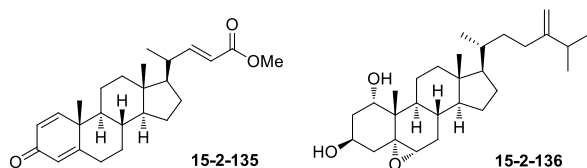
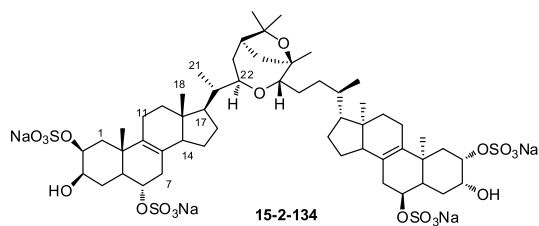
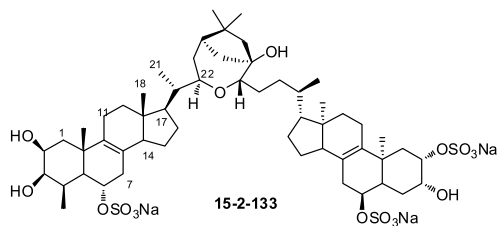
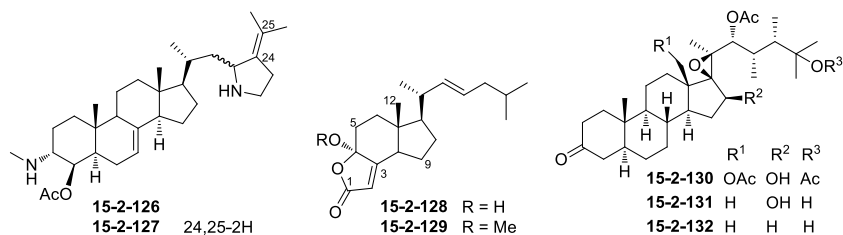
Table 15-2-39 (continued)

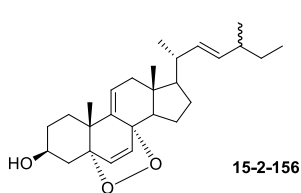
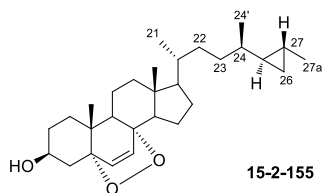
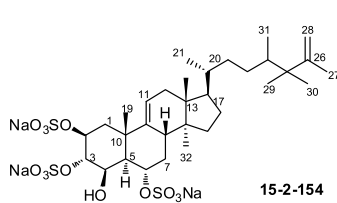
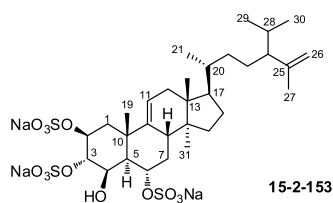
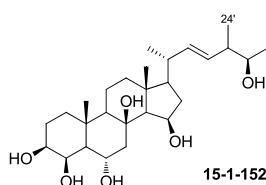
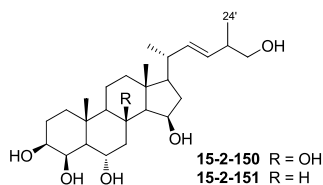
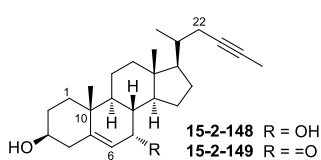
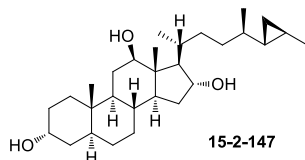
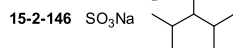
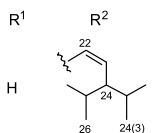
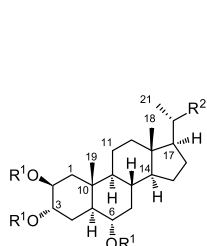
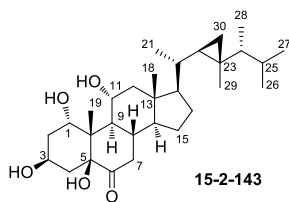
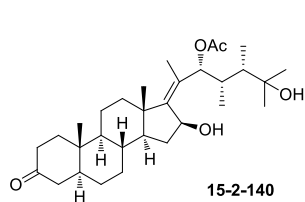
No.	Compounds	MFs	Test solvents	References
15-2-132	hippuristerone I	C ₃₁ H ₅₀ O ₅	CDCl ₃	[74]
15-2-133	amaroxocane A	C ₅₅ H ₈₅ Na ₃ O ₁₇ S ₃	CD ₃ OD	[75]
15-2-134	amaroxocane B	C ₅₄ H ₈₂ Na ₄ O ₂₀ S ₄	CD ₃ OD	[75]
15-2-135	methyl 3-oxochola-4,22-dien-24-oate	C ₂₅ H ₃₄ O ₃	CDCl ₃	[53]
15-2-136	gibberoepoxysterol	C ₂₈ H ₄₆ O ₃	CDCl ₃	[60]
15-2-137	abeohyosterone	C ₂₇ H ₄₂ O ₅	DMSO- <i>d</i> ₆	[76]
15-2-138	hippuristerone J	C ₃₅ H ₅₄ O ₁₀	CDCl ₃	[77]
15-2-139	hippuristerone K	C ₃₃ H ₅₂ O ₇	CDCl ₃	[77]
15-2-140	hippuristerone L	C ₃₁ H ₅₀ O ₅	C ₆ D ₆	[77]
15-2-141	hippuristerol E	C ₃₁ H ₅₂ O ₅	CDCl ₃	[77]
15-2-142	hippuristerol F	C ₃₁ H ₅₂ O ₆	CDCl ₃	[77]
15-2-143	1 α ,3 β ,5 β ,11 α -tetrahydroxygorgostan-6-one	C ₃₀ H ₅₀ O ₅	CDCl ₃	[77]
15-2-144	topsentinol K	C ₃₀ H ₅₂ O ₃	CD ₃ OD	[78]
15-2-145	topsentinol L	C ₃₀ H ₅₂ O ₃	CD ₃ OD	[78]
15-2-146	topsentinol K trisulfate	C ₃₀ H ₄₉ Na ₃ O ₁₂ S ₃	CD ₃ OD	[78]
15-2-147	aragusterol G	C ₂₉ H ₅₀ O ₃	CDCl ₃	[45]
15-2-148	gelliusterol A	C ₂₅ H ₃₈ O ₂	CD ₃ OD	[46]
15-2-149	gelliusterol B	C ₂₅ H ₃₆ O ₂	CDCl ₃	[46]
15-2-150	–	C ₂₆ H ₄₄ O ₆	C ₅ D ₅ N-CD ₃ OD	[47]
15-2-151	certonardosterol B ₃	C ₂₆ H ₄₄ O ₅	CD ₃ OD	[47]
15-2-152	certonardosterol A ₄	C ₂₇ H ₄₆ O ₆	CD ₃ OD	[47]
15-2-153	spheciosterol sulfate B	C ₃₁ H ₄₉ Na ₃ O ₁₃ S ₃	CD ₃ OD	[79]
15-2-154	spheciosterol sulfate C	C ₃₂ H ₅₁ Na ₃ O ₁₃ S ₃	CD ₃ OD	[79]
15-2-155	topsentisterol A ₁	C ₂₉ H ₄₆ O ₃	CD ₃ OD	[65]
15-2-156	topsentisterol A ₃	C ₂₇ H ₄₀ O ₃	CD ₃ OD	[65]
15-2-157	topsentisterol B ₁	C ₂₉ H ₄₆ O ₃	CD ₃ OD	[65]
15-2-158	topsentisterol B ₂	C ₂₉ H ₄₆ O ₃	CD ₃ OD	[65]
15-2-159	topsentisterol B ₃	C ₂₉ H ₄₆ O ₃	CD ₃ OD	[65]
15-2-160	topsentisterol C ₁	C ₂₉ H ₄₆ O ₄	CD ₃ OD	[65]
15-2-161	topsentisterol C ₂	C ₂₉ H ₄₈ O ₄	CD ₃ OD	[65]
15-2-162	topsentisterol C ₄	C ₂₉ H ₄₈ O ₃	CD ₃ OD	[65]
15-2-163	topsentisterol D ₁	C ₂₉ H ₄₄ O ₂	CD ₃ OD	[65]
15-2-164	topsentisterol D ₂	C ₂₇ H ₄₀ O ₂	CD ₃ OD	[65]
15-2-165	(22S)-2 α ,3 α -diacetoxo-11 β -hydroxy-24-methyl-22,25-epoxy-5 α -furostan-18,20 β -lactone	C ₃₂ H ₄₆ O ₉	CDCl ₃	[80]
15-2-166	(22S)-2 α ,3 α -dihydroxy-11 β -hydroxy-24-methyl-22,25-epoxy-5 α -furostan-18,20 β -lactone	C ₂₈ H ₄₂ O ₇	CDCl ₃	[80]
15-2-167	(22S)-2 α -acetoxo-3 α -hydroxy-11 β -hydroxy-24-methyl-22,25-epoxy-5 α -furostan-18,20 β -lactone	C ₃₀ H ₄₄ O ₈	CDCl ₃	[80]

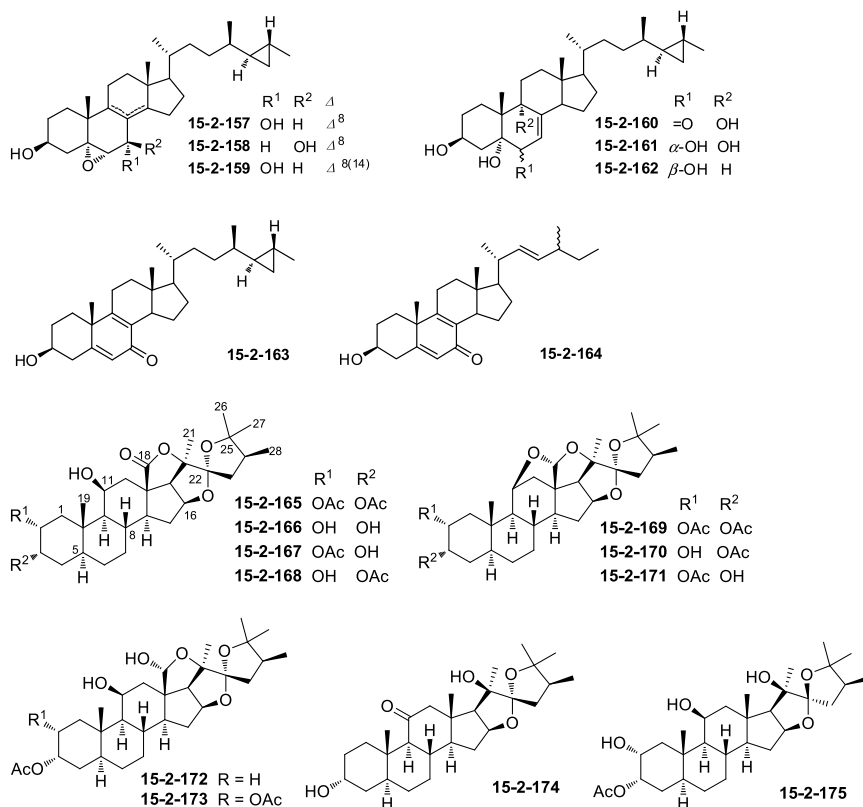
Table 15-2-39 (continued)

No.	Compounds	MFs	Test solvents	References
15-2-168	(22S)-2 α -hydroxy,3 α -acetoxy-11 β -hydroxy-24-methyl-22,25-epoxy-5 α -furostan-18,20 β -lactone	C ₃₀ H ₄₄ O ₈	CDCl ₃	[80]
15-2-169	(22S)-2 α ,3 α -diacetoxy-24-methyl-11 β ,18:18,20 β :22,25-triepoxy-5 α -furostane	C ₃₂ H ₄₆ O ₈	CDCl ₃	[80]
15-2-170	(22S)-2 α -hydroxy-3 α -acetoxy-24-methyl-11 β ,18:18,20 β :22,25-triepoxy-5 α -furostane	C ₃₀ H ₄₄ O ₇	CDCl ₃	[80]
15-2-171	(22S)-2 α -acetoxy,3 α -hydroxy-24-methyl-11 β ,18:18,20 β :22,25-triepoxy-5 α -furostane	C ₃₀ H ₄₄ O ₇	CDCl ₃	[80]
15-2-172	(22S)-3 α -acetoxy-11 β ,18 α -dihydroxy-24-methyl-18,20 β :22,25-diepoxy-5 α -furostane	C ₃₀ H ₄₆ O ₇	CDCl ₃	[80]
15-2-173	(22S)-2 α ,3 α -diacetoxy-11 β ,18 α -dihydroxy-24-methyl-18,20 β :22,25-diepoxy-5 α -furostane	C ₃₂ H ₄₈ O ₉	CDCl ₃	[80]
15-2-174	22- <i>epi</i> -hippuristan-11-one	C ₂₈ H ₄₄ O ₅	CDCl ₃	[80]
15-2-175	3-acetyl-2-desacetylhippurin-1	C ₃₀ H ₄₈ O ₇	CDCl ₃	[80]







**Table 15-2-40:** ^1H NMR spectroscopic data of marine steroids **15-2-118**–**15-2-120**.

H	15-2-118	15-2-119	15-2-120
1	2.18 m, 1.90 m	1.70 m, 1.36 m	ax 1.47 m, eq 1.86 m
2	2.54 m, 2.30 t(7.6)	1.92 m, 1.62 m	ax 1.46 m, eq 1.94 m
3		3.92 m	3.61 m
4	6.18 s	2.08 m, 1.31 m	ax 1.46 m, eq 2.31 m
5			1.78 m
6		2.92 d(4.5)	4.30 d(9.9)
7	2.68 dd(16.0, 4.0), 2.06 m	1.93 m, 1.50 m	6.60 d(1.7)
8	1.90 m	1.38 m	
9	1.40 m	1.26 m	
11	1.65 m, 1.51 m	1.37 m, 1.25 m	9.91 dd(1.7, 3.7)
12	2.11 m, 1.28 m	1.95 m, 1.10 m	2.04 dd(1.7, 15.7) 2.25 dd(3.7, 15.7)
14	1.20 m	1.17 m	3.56 t(10.0)
15	1.63 m, 1.18 m	1.55 m, 1.13 m	1.50 m, 1.77 m
16	1.90 m, 1.33 m	1.82 m, 1.26 m	1.66 m

Table 15-2-40 (continued)

H	15-2-118	15-2-119	15-2-120
17	1.20 m	1.20 m	1.90 m
18	0.73 s	0.61 s	0.73 s
19	1.17 s	1.07 s	1.11 s
20	1.38 m	1.33 m	2.15 m
21	0.94 d(6.8)	0.90 d(6.5)	1.00 d(6.5)
22	1.50 m, 1.22 m	1.46 m, 1.19 m	5.29 dd(6.3, 15.4)
23	1.36 m, 1.25 m	1.34 m, 1.23 m	5.25 dd(8.1, 15.4)
24	0.61 m	0.59 m	
25	0.15 m	0.14 m	2.22 m
26	0.46 m	0.45 m	0.95 d(6.8)
27	0.14 m, 0.07 m	0.14 m, 0.08 m	0.95 d(6.8)
28	1.02 d(6.4)	1.00 d(6.0)	
29	0.90 d(6.4)	0.88 d(6.5)	

Table 15-2-41: ^1H NMR spectroscopic data of marine steroids **15-2-121~15-2-123**.

H	15-2-121	15-2-122	15-2-123
1	7.05 d(10.2)	7.26 d(10.2)	7.05 d(10.1)
2	6.22 dd(10.2, 1.9)	6.26 dd(10.2, 1.9)	6.23 dd(10.1, 1.8)
4	6.07 bt(1.6)	6.00 bt(1.6)	6.06 bt(1.5)
6 α	2.35 ddd(13.3, 4.2, 2.6)	2.31 ddd(13.3, 4.9, 1.8)	2.35 ddd(13.2, 4.3, 2.8)
6 β	2.46 dddd(13.5, 13.3, 5.0, 1.4)	2.55 dddd(13.7, 13.5, 5.4, 1.5)	2.46 dddd(13.5, 13.5, 5.2, 1.5)
7 α	1.05 m	1.04 m	1.02 m
7 β	1.94 m	2.04 m	1.94 m
8	1.60 m	2.02 m	1.62 m
9	1.08 m	1.04 m	1.04 m
11	1.68 m	4.34 br s	1.68 m
12 α	1.18 m	1.44 m	1.20 m
12 β	2.00 ddd(12.9, 3.6, 3.2)	2.08 m	2.13 ddd(12.9, 3.7, 3.3)
14	1.00 m	0.96 m	0.98 m
15	1.10 m, 1.56 m	1.15 m, 1.58 m	1.14 m, 1.64 m
16	1.25 m, 1.63 m	1.28 m, 1.64 m	1.64 m
17	1.15 m	1.08 m	1.50 dd(9.5, 9.5)
18	0.75 s	0.97 s	0.87 s
19	1.23 s	1.45 s	1.22 s
20	2.06 m	1.98 m	
21	1.00 d(6.6)	0.99 d(6.7)	1.30 s
22	5.34 dd(15.3, 8.2)	5.13 ddd(15.2, 8.3, 1.3)	5.51 d(15.7)
23	5.43 dd(15.3, 7.4)	5.27 dd(15.2, 6.4)	5.48 dd(15.7, 5.5)
25	3.06 quint(7.1)	2.18 hept d(6.7, 5.8)	2.27 hept d(6.8, 5.5)
26		0.93 d(6.7)	0.98 d(6.8)
27	1.22 d(7.0)	0.93 d(6.7)	0.98 d(6.8)
OMe	3.67 s		

Table 15-2-42: ¹H NMR spectroscopic data of marine steroids **15-2-124~15-2-128**.

H	15-2-124	15-2-125	15-2-126	15-2-127	15-2-128
1	1.74 m 1.30 td(14.5, 3)	2.28 dd(18, 6) 2.02 m	1.39 m, 1.58 m	1.81 m 1.32 m	
2	2.12 dt(16, 3) 1.88 m	6.05 br m	1.88 m	2.11 m, 1.87 m	5.67 d(1.8)
3	3.31 m ^①	5.60 br d(5)	2.60 br d(3)	3.31 m ^①	
4	1.94 m	3.58 m	4.96 br s	5.09 br s	
5	1.60 m	1.71 m	1.82 m	1.76 m	2.23 ddd(14.0, 4.0, 2.5) 1.84 m
6	1.80 m, 1.88 m	1.92 m, 2.35 m	1.64 m, 2.01 m	1.81 m, 2.07 m	1.98 ddd(13.0, 5.0, 2.5) 1.62 m
7	5.32 br d(3.5)	5.25 br d(4.5)	5.22 br d(4)	5.27 br d(4)	
8					2.66 ddd(12.0, 7.0, 1.8)
9	1.98 m	1.92 m	1.82 m	1.83 m	1.73 m, 1.62 m
10					1.92 m, 1.49 m
11	1.62 m	1.62 m	1.58 m	1.58 m	1.50 m
12	1.40 m, 2.02 m	1.35 m, 2.13 dt(12.5, 3)	1.28 m, 2.20 m	1.35 m, 2.13 m	0.68 s
13					2.08 m
14	1.98 m	1.92 m	1.85 m	1.86 m	1.05 d(7.0)
15	2.38 ddd(8, 6, 3) 1.61 m	1.59 m	1.54 m	1.53 m	5.24 dd(15.0, 9.5)
16	3.77 dt(10, 8)	2.02 m, 1.35 m	1.90 m	1.96 m	5.36 dt(15.0, 8.5)
17	1.62 m	1.35 m	1.22 m	1.30 m	1.85 m
18	0.78 s	0.62 s	0.62 s	0.63 s	1.57 m
19	0.93 s	0.92 s	1.05 s	1.09 s	0.84 d(7.0)
20	1.68 m	1.54 m	1.56 m	1.49 m	0.88 d(7.0)
21	1.12 d(6)	1.08 d(6)	1.07 d(6.5)	1.07 d(6)	
22	2.23 ddd(12.5, 7.5, 2) 1.40 m	1.55 m, 1.81 m	1.04 m ^① , 1.54 m	1.46 m, 1.78 m	
23	4.49 br t(9)	3.17 m	3.84 br d(11)	3.31 m ^①	
24		2.02 m		1.83 m	
25		1.84 m		1.81 m	
26	1.73 br s	0.97 d(6.5)	1.66 br s	0.93 d(6.5)	
27	1.75 br s	1.01 d(6.5)	1.66 br s	1.01 d(6.5)	
28	2.73 m	2.02 m	2.36 m	2.07 m	
29	3.56 dt(12, 4) 3.35 m ^①	3.60 m 3.06 dt(11, 9)	3.01 dt(11, 7.5) 2.91 m	3.27 m	
NMe	2.92 s, 2.92 s	2.93 s	2.39 s, 2.06 s	2.80 s, 2.12 s	

^① Observed m.

Table 15-2-43: ^1H NMR spectroscopic data of marine steroids **15-2-129~15-2-132**.

H	15-2-129	15-2-130	15-2-131	15-2-132
1		2.00 m, 1.40 m	2.05 m, 1.33 m	2.04 m, 1.62 m
2	5.67 d(1.8)	2.38 m	2.30 m	2.32 m
4		2.30 m, 2.10 m	2.27 m, 2.10 m	2.27 m, 2.09 m
5	2.23 ddd(14.0, 4.0, 2.5) 1.84 m	1.56 m	1.56 m	1.54 m
6	1.98 ddd(13.0, 5.0, 2.5) 1.62 m	1.40 m	1.40 m	1.37 m
7		1.80 m, 0.92 m	1.82 m, 0.92 m	1.78 m, 0.92 m
8	2.66 ddd(12.0, 7.0, 1.8)	1.76 m	1.56 m	1.49 m
9	1.73 m, 1.62 m	0.89 m	0.70 m	0.75 m
10	1.92 m, 1.49 m			
11	1.50 m	1.52 m, 1.34 m	1.60 m, 1.48 m	1.59 m, 1.37 m
12	0.68 s	2.15 m, 1.40 m	1.82 m, 1.48 m	1.88 m, 1.46 m
13	2.08 m			
14	1.05 d(7.0)	1.42 m	1.06 m	1.29 m
15	5.24 dd(15.0, 9.5)	2.28 m, 1.45 m	2.20 m, 1.45 m	1.64 m, 1.40 m
16	5.36 m	4.05 t(6.9)	4.06 t(7.2)	2.08 m, 1.91 m
17	1.85 m			
18	1.57 m	4.31 d(11.4) 4.21 d(11.4)	0.95 s	0.92 s
19	0.84 d(7.0)	1.02 s	1.01 s	1.00 s
20	0.88 d(7.0)			
21		1.59 s	1.59 s	1.55 s
22		4.67 d(10.7)	4.62 d(10.8)	4.73 d(10.7)
23		2.30 m	2.45 m	2.33 m
24		2.05 m	1.55 m	1.59 m
26		1.56 s	1.23 s	1.28 s
27		1.42 s	1.20 s	1.20 s
28		0.90 d(7.4)	0.88 d(7.2)	0.91 d(7.4)
29		0.87 d(6.6)	0.84 d(6.9)	0.82 d(6.6)
OAc		2.12 s	2.11 s	2.08 s
OMe	3.21 m	2.07 s, 1.98 s		
OH			3.27 s(16-OH)	

Table 15-2-44: ^1H NMR spectroscopic data of marine steroids **15-2-133~15-2-135**.

H	15-2-133	15-2-134	15-2-135
1	1.85 dd(14.6, 3.0), 1.97 m	1.46 m, 2.45 dd(14.6, 3.1)	7.00 d(10)
2	4.16 br q(3.0)	4.72 br q(3.1)	6.19 dd(2, 10)
3	3.96 dd(6.1, 3.5)	4.01 dd(11.5, 4.3)	
4	2.50 m	1.64 m, 2.51 m	6.04 t(2)
5	2.02 dd(12.0, 4.1)	1.68 m	
6	4.87 dt(12.0, 7.6)	4.48 m	α 2.44 m, β 2.32 m

Table 15-2-44 (continued)

H	15-2-133	15-2-134	15-2-135
7	2.33 m, 2.91 dd(18.2, 6.8)	2.00 m, 2.70 m	α 1.00 m, β 1.90 m
8			1.55 m
9			0.98 m
11	2.10 m, 2.20 m	2.17 m, 2.22 m	α 1.65 m, β 1.57 m
12	1.36 m, 2.00 m	1.37 m, 2.04 m	α 0.99 m, β 1.74 m
14	1.94 m	2.26 m	0.95 m
15	1.38 m, 1.62 m	1.36 m, 1.58 m	α 1.62 m, β 1.14 m
16	1.29 m, 2.13 m	1.31 m, 2.15 m	α 1.86 m, β 1.29 m
17	1.37 m	1.51 m	1.23 m
18	0.64 s	0.65 s	0.69 s
19	1.27 s	1.24 s	1.19 s
20	1.24 m	1.33 m	2.24 m
21	0.95 d(6.7)	0.99 d(7.4)	0.96 d(6.7)
22	3.63 dd(11.8, 4.6)	3.67 dd(11.4, 4.3)	6.82 dd(10, 15.7)
23	1.16 m, 1.93 m	1.27 m, 2.03 m	5.75 d(15.7)
24	1.81 m	2.15 m	
26	1.03 s	1.32 s	
27	1.14 s	1.23 s	
29	1.28 d(6.6)		
1'	1.63 m, 2.32 dd(14.6, 3.2)	1.69 m, 2.36 dd(14.6, 3.1)	
2'	4.72 br q(2.6)	4.74 br q(3.1)	
3'	3.80 m	3.94 m	
4'	2.05 m, 1.60 m	2.16 m, 1.61 m	
5'	1.61 m	1.75 td(12.4, 2.2)	
6'	4.09 td(10.7, 5.2)	4.48 m	
7'	3.02 dd(13.6, 5.2), 1.92 m	2.07 m, 2.70 m	
9'	1.87 m		
11'	1.69 m, 1.56 m	2.17 m, 2.22 m	
12'	1.97 m, 1.13 m	1.37 m, 2.04 m	
14		2.23 m	
15'	2.35 m, 2.26 m	1.40 m, 1.67 m	
16'	1.83 m, 1.41 m	1.40 m, 1.90 m	
17'	1.29 m	1.29 m	
18'	0.86 s	0.68 s	
19'	0.91 s	1.22 s	
20'	1.68 m	1.65 m	
21'	1.00 d(6.6)	0.98 d(7.4)	
22'	1.51 m, 1.68 m	1.51 m, 2.03 m	
23'	1.39 m, 1.79 m	1.42 m, 1.51 m	
24'	3.26 d(7.9)	3.16 d(6.6)	
26'	1.93 m, 2.05 m	2.05 m, 2.26 m	
27'	1.62 m, 1.76 m	1.15 s	
OAc			3.71 s

Table 15-2-45: ^1H NMR spectroscopic data of marine steroids **15-2-136~15-2-139**.

H	15-2-136	15-2-137	15-2-138	15-2-139
1	3.89 br s($W_{1/2}$ 8.5)	α 1.75 m, β 1.40 m	2.05 m, 1.33 m	2.00 m, 1.32 m
2	α 2.23 m β 1.82 ddd(12.0, 12.0, 2.5)	3.69 m	2.30 m	2.34 m
3	4.29 m	3.97 m		
4	α 1.37 ddd(13.5, 4.0, 2.0) β 2.34 dd(13.5, 12.0)	6.06 d(4.0)	2.28 m, 2.11 m	2.27 m, 2.08 m
5			1.58 m	1.52 m
6	2.84 d(4.5)		1.40 m	1.36 m
7	α 1.47 dd(15.0, 10.5) β 1.92 m	α 2.28 d(16.5) β 2.45 d(16.5)	1.82 m 0.92 m	1.78 m 0.94 m
8	1.41 m		1.56 m	1.52 m
9	1.69 ddd(12.0, 12.0, 5.0)	2.94 dd(10.4, 6.1)	0.70 m	0.74 m
11	α 1.50 m β 1.29 ddd(12.0, 12.0, 4.0)	α 2.00 m β 1.81 m	1.63 m 1.42 m	1.60 m 1.38 m
12	α 1.18 ddd(13.0, 12.0, 4.0) β 1.97 ddd(13.0, 4.0, 3.0)	α 1.53 m β 1.67 m	1.42 m 1.84 m	1.38 m 1.79 m
14	0.99 m		1.07 m	1.23 m
15	α 1.57 m, β 0.99 m	α 2.57 m, β 2.13 m	2.20 m, 1.46 m	1.64 m, 1.34 m
16	α 1.84 m, β 1.25 m	α 1.78 m, β 1.57 m	4.12 t(7.2)	2.10 m, 1.87 m
17	1.11 m	1.56 m		
18	0.63 s	0.79 s	1.00 s	0.92 s
19	1.09 s	1.02 s	1.02 s	1.01 s
20	1.38 m	1.52 m		
21	0.93 d(6.5)	0.97 d(6.8)	5.02 d(12.6) 4.38 d(12.6)	1.56 s
22	1.14 m, 1.53 m	1.36 m, 0.85 m	4.74 d(10.5)	4.74 d(10.5)
23	1.86 m 2.09 ddd(15.5, 11.5, 5.0)	1.34 m 1.34 m	2.45 m	2.43 m
24		1.34 m, 1.21 m	1.70 m	1.77 m
25	2.21 m			
26	1.02 d(7.0)	1.05 s	4.07 d(11.2) 4.03 d(11.2)	3.92 d(11.5) 4.04 d(11.5)
27	1.02 d(7.0)	1.05 s	1.26 s	1.17 s
28	4.65 s, 4.71 s		0.95 d(7.4)	0.90 d(7.5)
29			0.85 d(6.6)	0.85 d(6.5)
OAc			2.11 s, 2.11 s, 2.11 s	2.10 s, 2.13 s
OH	2.35 d(8.5, 1-OH)	4.24 (2-OH) 4.83 (3-OH) 4.04 (25-OH)		

Table 15-2-46: ^1H NMR spectroscopic data of marine steroids **15-2-140~15-2-143**.

H	15-2-140	15-2-141	15-2-142	15-2-143
1	1.48 m, 0.83 m	1.50 m, 1.29 m	1.45 m, 1.30 m	4.34 dd(13.0 3.5)
2	2.18 m	1.62 m	1.66 m	2.40 dt(13.5, 3.0) 2.06 m
3		4.06 br s	4.06 br s	4.20 br s
4	1.88 m, 1.97 m	1.40 m, 1.56 m	1.38 m, 1.52 m	1.65 br d(14.5) 2.31 dd(14.0, 3.5)
5	1.05 m	1.55 m	1.53 m	
6	0.93 m	1.24 m	1.24 m	
7	1.44 m	1.78 m	1.76 m	2.47 dd(14.0, 4.5)
	0.53 m	0.95 m	0.94 m	2.32 t(14.0)
8	1.22 m	1.42 m	1.51 m	1.79 m
9	0.35 m	0.72 m	0.73 m	1.98 br t(10.0)
11	1.28 m	1.64 m, 1.26 m	1.61 m, 1.27 m	3.96 m
12	1.41 m, 2.21 m	1.35 m, 1.78 m	1.34 m, 1.79 m	1.34 m, 2.44 dd (14.0, 4.5)
14	0.96 m	1.24 m	1.06 m	1.37 m
15	2.31 m, 1.63 m	1.67 m, 1.31 m	2.22 m, 1.38 m	1.61 m
16	5.06 t(7.0)	2.08 m, 1.88 m	4.05 t(7.5)	1.37 m, 2.12 m
17				1.36 m
18	1.18 s	0.91 s	0.93 s	0.64 s
19	0.56 s	0.78 s	0.78 s	1.05 s
20				1.05 m
21	1.81 s	1.57 s	1.59 s	1.05 s
22	5.56 d(10.5)	4.75 d(10.7)	4.61 d(11.0)	0.17 m
23	2.33 m	2.37 m	2.44 m	
24	1.61 m	1.64 m	1.55 m	0.25 m
25				1.57 m
26	0.95 s	1.27 s	1.25 s	0.86 d(6.5)
27	1.01 s	1.21 s	1.20 s	0.95 d(6.5)
28	0.75 d(7.0)	0.93 d(7.0)	0.89 d(7.5)	0.93 d(7.0)
29	0.91 d(6.5)	0.84 d(7.0)	0.85 d(7.0)	0.90 s
30				-0.11 dd(4.5, 5.5) 0.49 dd(4.5, 9.5)
OH	5.37 s(30-OH)			4.63 s(5-OH)
OAc	1.59 s	2.09 s	2.12 s	

Table 15-2-47: ^1H NMR spectroscopic data of marine steroids **15-2-144~15-2-146**.

H	15-2-144	15-2-145	15-2-146
1	1.70 dd(13.6, 2.0) 1.40 dd(13.6, 3.4)	1.68 dd(13.5, 2.5) 1.41 dd(13.5, 3.7)	2.07 br d(14.5) 1.46 dd(15.0, 2.5)
2	3.79 br s	3.79 br s	4.79 br s
3	3.81 br d(2.3)	3.81 br d(2.5)	4.75 br s

Table 15-2-47 (continued)

H	15-2-144	15-2-145	15-2-146
4	1.69 m 1.93 ddd(12.5, 8.5, 3.0)	1.68 br s 1.93 ddd(12.6, 9.2, 3.5)	2.28 br d(15.0) 1.79 ddd(15.0, 13.0, 2.0)
5	1.42 m	1.44 m	1.62 ddd(14.0, 10.5, 2.0)
6	3.35 m	3.35 ddd(10.9, 10.9, 4.5)	4.18 ddd(10.5, 10.5, 4.5)
7	0.87 m, 1.93 m	0.86 m, 1.94 m	1.02 m, 2.35 ddd(12.0, 4.5, 4.5)
8	1.48 m	1.47 m	1.52 m
9	0.69 m	0.68 m	0.75 m
11	1.51 m, 1.30 m	1.53 m, 1.30 m	1.54 m, 1.32 m
12	1.99 m, 1.16 m	1.99 m, 1.16 m	2.00 m, 1.17 m
14	1.08 m	1.09 m	1.11 m
15	1.58 m, 1.06 m	1.58 m, 1.08 m	1.57 m, 1.10 m
16	1.70 m, 1.30 m	1.70 m, 1.29 m	1.71 m, 1.21 m
17	1.24 m	1.20 m	1.20 m
18	0.71 s	0.70 s	0.72 s
19	0.98 s	0.99 s	1.04 s
20	2.41 m	2.07 m	2.42 m
21	0.98 d(6.7)	1.02 d(6.8)	0.98 d(6.5)
22	5.28 t(10.8)	5.14 dd(15.3, 8.4)	5.28 t(11.0)
23	4.97 t(10.8)	4.97 dd(15.3, 9.5)	4.98 t(11.0)
24	1.86 m	1.44 m	1.91 m
25	1.76 m	1.70 m	1.63 m
26	0.80 d(6.6)	0.78 d(6.7)	0.88 d(6.5)
27	0.91 d(6.6)	0.86 d(6.7)	0.92 d(6.5)
24(1)	1.73 m	1.70 m	1.61 m
24(2)	0.84 d(6.6)	0.79 d(6.8)	0.80 (6.6)
24(3)	0.89 d(6.6)	0.85 d(6.8)	0.90 d(6.6)

Table 15-2-48: ^1H NMR spectroscopic data of marine steroids **15-2-147~15-2-149**.

H	15-2-147	15-2-148	15-2-149
1		1.13 m, 1.84 m	1.15 m, 1.95 m
2		1.47 m, 1.78 m	1.60 m, 1.93 m
3	4.00 m	3.47 m	3.68 m
4		2.25 m	2.40 m 2.49 ddd(1.9, 4.8, 14.1)
6		5.53 d(5.0)	5.69 br s
7		3.75 br s	
8		1.44 m	2.22 m
9		1.30 m	1.52 m
11		1.53 m	1.58 m
12	3.53 dd(4.6, 10.9)	1.15 m 2.00 dt(2.9, 12.7)	1.15 m 2.02 dt(3.0, 12.9)
14		1.45 m	1.35 m

Table 15-2-48 (continued)

H	15-2-147	15-2-148	15-2-149
15		1.10 m, 1.78 m	1.24 m, 2.41 m
16	4.32 t(6.8)	1.26 m, 1.85 m	1.25 m, 1.87 m
17		1.27 m	1.15 m
18	0.71 s	0.71 s	0.69 s
19	0.78 s	1.00 s	1.20 s
20	1.84 m	1.50 m	1.51 m
21	1.09 d(6.9)	1.06 d(6.5)	1.07 d(6.5)
22		1.95 m	1.95 m
		2.14 dt(2.5, 16.5)	2.19 m
24	0.65 m		
25	0.15 m	1.73 t(2.4)	1.79 br t(2.2)
26	0.46 m		
27	0.09 m, 0.15 m		
28	0.91 d(6.7)		
29	1.00 d(6.0)		

Table 15-2-49: ¹H NMR spectroscopic data of marine steroids 15-2-150~15-2-152.

H	15-2-150	15-2-151	15-2-152
1	1.91 dt(13.0, 3.5) 1.17 m	1.68 dt(13.5, 3.5) 1.04 m	1.70 dt(13.0, 3.5) 0.98 m
2	2.39 m, 1.98 m	1.78 m, 1.56 m	1.83 m, 1.55 m
3	3.96 ddd(12.0, 5.0, 3.5)	3.42 ddd(12.0, 4.8, 3.5)	3.42 ddd(11.5, 4.8, 3.3)
4	5.25 br s	4.21 br s	4.25 br s
5	1.46 dd(11.0, 2.5)	0.91 m	0.94 m
6	5.07 td(10.8, 4.5)	3.89 td(11.0, 4.5)	4.15 td(11.0, 4.0)
7	3.16 dd(12.0, 4.0) 1.86 m	2.34 dt(11.5, 4.3) 0.92 m	2.43 dd(12.0, 4.5) 1.29 m
8		1.88 m	
9	1.04 dd(12.5, 2.5)	0.67 td(11.8, 3.5)	0.82 dd(12.0, 3.0)
11	2.13 qd(13.0, 3.0) 1.62 dd(13.0, 3.0)	1.44 m 1.31 m	1.77 m 1.43 m
12	2.08 dt(12.0, 3.3) 1.25 m	1.92 m 1.11 m	1.96 dt(12.5, 3.3) 1.16 m
14	1.16 m	0.89 m	1.01 d(5.5)
15	4.71 br t(6.2)	4.13 td(6.5, 2.5)	4.38 td(6.5, 2.5)
16	2.36 m 1.74 ddd(14.3, 11.0, 2.3)	2.20 m 1.33 m	2.18 dt(14.5, 8.3) 1.34 m
17	1.06 m	1.11 m	1.03 m
18	1.66 s	0.94 s	1.27 s
19	1.86 s	1.06 s	1.15 s
20	2.31 m	2.14 m	2.14 m
21	1.14 d(6.5)	1.01 d(6.5)	0.990 d(7.0)

Table 15-2-49 (continued)

H	15-2-150	15-2-151	15-2-152
22	5.48 dd(15.5, 8.0)	5.30 dd(15.0, 8.0)	5.26 dd(15.0, 8.0)
23	5.54 dd(15.5, 6.5)	5.27 dd(15.0, 7.0)	5.23 dd(15.0, 7.0)
24	2.56 m(6.5)	2.22 m	2.01 q(6.5)
25	3.84 dd(10.0, 5.5)	3.42 dd(11.0, 6.5)	3.46 quint(6.8)
	3.70 dd(10.0, 7.0)	3.30 m	
26			1.10 d(6.5)
24'	1.23 d(7.0)	0.97 d(7.0)	0.99 d(7.0)

Table 15-2-50: ^1H NMR spectroscopic data of marine steroids **15-2-153~15-2-156**.

H	15-2-153	15-2-154	15-2-155	15-2-156
1	α 1.86 br dd(14.7, 3.9) β 2.31 br d(14.7)	α 1.83 br dd(14.6, 3.9) β 2.37 br d(14.6)	1.70 dt(13.5, 3.0) 1.85 m	2.00 m
2	4.96 m	4.99 m	1.52 m, 1.77 m	1.55 m, 1.85 m
3	4.82 (ov)	4.82 (ov)	3.77 m	3.83 m
4	4.46 dt(2.8, 1.0)	4.47 dt(3.0, 1.0)	1.88 m, 2.00 m	1.95 m
5	1.48 dd(11.4, 2.8)	1.50 dd(11.3, 3.0)		
6	4.84 (ov)	4.84 (ov)	6.25 d(8.5)	6.33 d(8.0)
7	1.55 m 2.23 dt(11.7, 4.5)	1.57 m 2.24 dt(11.8, 4.6)	6.53 d(8.5)	6.67 d(8.0)
8	2.49 br d(12.9)	2.51 br d(12.9)		
9			1.44 m	
11	5.35 br d(5.2)	5.35 br d(5.4)	1.51 m	5.50 dd(6.5, 2.0)
12	2.11 br d(16.8) 1.95 dd(16.8, 5.2)	2.10 br d(17.1) 1.98 dd(16.9, 5.5)	1.22 m 1.98 m	2.05 m 2.32 dd(17.0, 6.5)
14			1.49 m	1.82 m
15	α 1.36 m, β 1.45 m	α 1.39 m, β 1.47 m	1.24 m, 1.52 m	1.24 m, 1.55 m
16	α 1.89 m, β 1.31 m	α 1.92 m, β 1.33 m	1.40 m, 1.94 m	1.38 m
17	1.63 m	1.68 m	1.20 m	1.36 m
18	0.68 s	0.70 s	0.84 s	0.79 s
19	1.42 s	1.45 s	0.89 s	1.12 s
20	1.34 m	1.38 m	1.35 m	2.05 m
21	0.91 d(6.2)	0.90 br d(6.5)	0.93 d(7.0)	1.03 d(6.5)
22	1.38 m 0.97 m	1.43 m 1.07 dd(13.3, 6.6)	1.25 m	5.21 m
23	1.53 m, 1.08 m	1.57 m, 1.32 m	1.37 m	5.18 m
24	1.52 m	1.39 m	0.58 m	1.92 m
25			0.13 m	1.18 m, 1.25 m
26	4.77 dd(2.5, 1.4) 4.62 br d(2.5)		0.09 m 0.15 m	0.88 t(7.0)
27	1.57 br s	1.70 br s	0.45 m	

Table 15-2-50 (continued)

H	15-2-153	15-2-154	15-2-155	15-2-156
28	1.52 m	4.74 dd(2.4, 1.3) 4.72 br d(2.4)		
29	0.81 d(6.2)	0.97 s		
30	0.92 d(6.2)	0.96 s		
31	0.80 s	0.82 d(6.7)		
32		0.81 s		
27a			1.01 d(6.0)	
24'			0.91 d(7.0)	0.96 d(6.5)

Table 15-2-51: ¹H NMR spectroscopic data of marine steroids 15-2-157~15-2-159.

H	15-2-157	15-2-158	15-2-159
1	1.58 m 1.78 m	1.58 m 1.78 m	1.68 ddd(12.5, 3.5, 2.5) 1.96 ddd(12.5, 4.0, 2.0)
2	1.55 m, 1.90 m	1.62 m, 1.90 m	1.55 m, 1.90 m
3	3.75 m	3.74 m	3.75 m
4	1.35 m 2.13 m	1.37 m 2.16 dd(13.0, 11.5)	1.32 m 2.11 dd(13.0, 12.0)
6	3.20 br s	3.02 d(3.0)	3.05 d(3.0)
7	4.25 br s	4.28 d(3.0)	4.42 d(3.0)
9			2.41 m
11	2.05 m	2.05 m	1.45 m
12	1.18 m	1.18 m	1.20 m, 1.93 m
14	2.16 m	2.12 m	
15	1.90 m	1.65 m	2.28 m, 2.68 ddd(12.5, 8.5, 8.0)
16	1.47 m	1.38 m, 1.98 m	1.45 m, 1.90 m
17	1.16 m	1.15 m	1.24 m
18	0.62 s	0.66 s	0.89 s
19	1.13 s	1.28 s	0.87 s
20	1.46 m	1.38 m	1.46 m
21	0.95 d(6.5)	0.95 d(7.0)	0.95 d(6.5)
22	1.40 m, 1.56 m	1.45 m, 1.55 m	1.24 m, 1.46 m
23	1.28 m, 1.35 m	1.28 m, 1.35 m	1.28 m, 1.35 m
24	0.60 m	0.60 m	0.60 m
25	0.13 m	0.12 m	0.13 m
26	0.09 m, 0.15 m	0.09 m, 0.15 m	0.09 m, 0.15 m
27	0.45 m	0.45 m	0.45 m
24'	0.91 d(7.0)	0.90 d(6.5)	0.90 d(6.5)
27a	1.01 d(6.0)	1.01 d(6.5)	1.01 d(6.0)

Table 15-2-52: ^1H NMR spectroscopic data of marine steroids **15-2-160** and **15-2-161**.

H	15-2-160	15-2-161	H	15-2-160	15-2-161
1	1.52 m 2.29 td(13.5, 3.5)	1.38 m 2.22 m	17	1.41 m	1.33 m
2	1.45 m, 1.86 m	1.44 m, 1.85 m	18	0.65 s	0.60 s
3	3.92 m	3.89 m	19	0.99 s	1.05 s
4	1.63 dd(14.0, 11.5) 2.02 dd(14.0, 2.0)	1.46 m 2.12 m	20	1.38 m	1.34 m
6		3.87 br s	21	0.97 d(7.0)	0.95 d(6.5)
7	5.58 d(2.0)	5.05 br s	22	1.23 m, 1.52 m	1.28 m
11	1.74 m, 1.95 m	1.28 m, 2.12 m	23	1.30 m, 1.38 m	1.38 m
12	1.28 m, 1.56 m	1.58 m, 1.82 m	24	0.62 m	0.60 m
14	2.74 ddd(12.0, 7.0, 2.0)	2.49 m	25	0.11 m	0.12 m
15	1.52 m, 1.69 m	1.55 m	26	0.09 m, 0.15 m	0.09 m, 0.15 m
16	2.01 m	1.55 m, 1.82 m	27	0.46 m	0.45 m
			27a	1.01 d(7.0)	1.01 d(6.5)
			24'	0.91 d(6.5)	0.91 d(7.0)

Table 15-2-53: ^1H NMR spectroscopic data of marine steroids **15-2-162~15-2-164**.

H	15-2-162	15-2-163	15-2-164
1	1.45 m, 1.55 m	1.25 m, 2.15 m	1.25 m, 2.15 m
2	1.77 m	1.71 ddd(25.0, 13.5, 4.0), 1.92 m	1.71 ddd(25.0, 13.5, 4.5), 1.91 m
3	3.96 m	3.54 m	3.54 m
4	1.67 ddd (13.5, 5.0, 2.0) 2.05 m	2.55 m	2.55 m
6	3.54 m	6.00 br s	6.00 br s
7	5.26 m		
9	1.97 m		
11	1.55 m	2.50 m	2.50 m
12	1.30 m, 2.05 m	1.51 m, 2.14 m	1.51 m, 2.12 m
14	1.94 m	2.25 m	2.26 m
15	1.52 m, 1.57 m	1.40 m, 2.50 m	1.39 m, 2.52 m
16	1.42 m	1.37 m, 2.02 m	1.36 m, 1.82 m
17	1.25 m	1.26 m	1.26 m
18	0.63 s	0.67 s	0.68 s
19	1.05 s	1.37 s	1.38 s
20	1.35 m	1.40 m	2.06 m
21	0.95 d(6.0)	0.99 d(7.0)	1.06 d(7.0)
22	1.24 m	1.05 m, 1.57 m	5.22 dd(15.0, 8.0)
23	1.33 m	1.27 m, 1.38 m	5.16 dd(15.0, 7.5)
24	0.60 m	0.60 m	1.92 m
24'	0.91 d(7.0)	0.91 d(7.0)	0.94 d(6.5)
25	0.11 m	0.13 m	1.24 m, 1.32 m
26	0.09 m, 0.15 m	0.09 m, 0.15 m	0.86 t(7.0)
27	0.45 m	0.45 m	
27a	1.01 d(6.5)	1.01 d(6.5)	

Table 15-2-54: ¹H NMR spectroscopic data of marine steroids **15-2-165**–**15-2-169**.

H	15-2-165	15-2-166	15-2-167	15-2-168	15-2-169
1	1.58 m, 1.83 m	1.40 t(12.0)	1.56 m	1.35 t(12.1), 2.06 m	1.58 m, 1.80 m
		1.94 dd(12.0, 5.0)	1.92 dd(11.5, 4.5)		
2	5.02 br d(11.4)	3.86 br d(12.0)	5.04 br d(12.0)	3.92 br d(12.2)	5.02 br d(11.1)
3	5.28 br d(2.2)	3.97 br s	4.06 br s	5.14 br d(2.3)	5.29 br d(2.6)
4	1.53 m, 1.36 m	1.54 m	2.35 m, 1.59 m	1.59 m	1.55 m
5	1.49 m	1.50 m	1.59 m	1.52 m	1.53 m
6	1.28 m	1.27 m	1.27 m	1.25 m	1.26 m
7	1.95 m, 0.88 m	1.85 m, 0.89 m	1.85 m, 0.91 m	1.85 m, 0.88 m	1.78 m, 1.10 m
8	2.50 m	2.48 m	2.50 m	2.50 m	1.70 m
9	0.90 m	0.93 m	0.94 m	0.94 m	0.85 d(10.8)
11	4.13 dd(12.6, 3.3)	4.20 br d(12.0)	4.15 dd(13.0, 2.5)	4.20 br d(12.6)	4.77 br d(5.4)
12	2.40 m	1.70 dd(14.0, 4.0)	1.68 dd(14.0, 4.0)	1.71 dd(14.0, 3.6)	2.30 m
	1.71 m	2.37 dd(14.0, 2.0)	2.36 dd(14.0, 2.0)	2.38 br d(14.2)	1.40 m
14	1.51 m	1.49 m	1.50 m	1.50 m	1.75 m
15	2.26 m, 1.37 m	2.32 m, 1.23 m	2.31 m, 1.23 m	2.31 m, 1.23 m	2.22 m, 1.37 m
16	4.66 dt(6.6, 6.6)	4.68 dt(8.0, 7.5)	4.66 dt(8.0, 7.0)	4.67 dt(7.4, 6.9)	4.58 dt(6.8, 1.7)
17	2.66 d(8.6)	2.67 d(8.5)	2.66 d(8.0)	2.67 d(8.3)	2.61 d(6.9)
18					5.31 s
19	1.17 s	1.11 s	1.16 s	1.13 s	0.91 s
21	1.47 s	1.48 s	1.48 s	1.48 s	1.37 s
23	2.11 m	2.12 dd(13.0, 6.0)	2.12 dd(13.0, 6.5)	2.12 m	2.05 m
	1.79 m	1.80 t(13.0)	1.80 t(13.0)	1.80 t(13.0)	1.75 m
24	2.30 m	2.31 m	2.30 m	2.30 m	2.25 m
26	1.29 s	1.30 s	1.30 s	1.30 s	1.28 s
27	0.99 s	1.00 s	1.00 s	1.00 s	0.98 s
28	0.96 d(6.8)	0.96 d(6.5)	0.96 d(6.5)	0.97 d(6.9)	0.94 d(6.8)
11-OH	3.95 d(12.6)	4.00 d(12.5)	4.00 d(12.5)	3.98 d(12.7)	
OAc	2.04 s, 1.97 s		2.01 s	2.10 s	2.04 s, 1.98 s

Table 15-2-55: ¹H NMR spectroscopic data of marine steroids **15-2-170**–**15-2-172**.

H	15-2-170	15-2-171	15-2-172
1	1.20 t(12.0)	1.41 t(12.0)	1.34 m
	1.94 dd(12.0, 4.5)	1.80 m	1.70 m
2	3.92 br d(12.0)	5.01 br d(12.0)	1.70 m
3	5.13 br d(3.0)	4.05 br d(2.0)	5.01 br s
4	1.50 m	1.54 m	1.46 m

Table 15-2-55 (continued)

H	15-2-170	15-2-171	15-2-172
5	1.33 m	1.54 m	1.47 m
6	1.24 m	1.36 m	1.18 m
7	1.80 m, 1.06 m	1.78 m, 1.07 m	1.82 m, 0.94 m
8	1.65 m	1.64 m	1.88 m
9	0.88 br d(11.0)	0.90 br d(11.0)	0.81 br d(11.1)
11	4.82 br d(5.5)	4.76 br d(5.5)	4.29 br s
12	2.30 dd(11.5, 5.5)	2.28 dd(10.5, 5.5)	2.71 d(12.0)
	1.38 br d(11.0)	1.37 br d(10.0)	1.68 m
14	1.75 m	1.74 m	1.48 m
15	2.35 m, 1.53 m	2.34 m, 1.56 m	2.11 m, 1.30 m
16	4.59 dt(7.0, 2.0)	4.58 dt(7.0, 2.0)	4.53 dt(7.9, 7.2)
17	2.62 d(6.5)	2.61 d(7.0)	2.62 d(8.0)
18	5.31 s	5.31 s	5.32 s
19	0.94 s	0.97 s	1.02 s
21	1.37 s	1.37 s	1.43 s
23	2.04 dd(13.5, 6.5)	2.05 dd(13.0, 6.5)	2.09 m
23	1.72 m	1.74 m	1.72 m
24	2.24 m	2.25 m	2.28 m
26	1.28 s	1.28 s	1.30 s
27	0.98 s	0.97 s	0.97 s
28	0.94 d(6.5)	0.94 d(7.0)	0.94 d(6.9)
OAc	2.10 s	2.08 s	2.04 s

Table 15-2-56: ^1H NMR spectroscopic data of marine steroids 15-2-173~15-2-175.

H	15-2-173	15-2-174	15-2-175
1	1.50 m, 1.87 m	1.21 m, 2.27 m	1.31 m, 1.98 m
2	5.00 br d(11.3)	1.74 m, 1.54 m	3.90 m
3	5.28 br s	4.04 br s	5.12 br s
4	1.54 m	1.53 m, 1.37 m	1.55 m, 1.57 m
5	1.47 m	1.52 m	1.53 m
6	1.26 m	1.19 m	1.26 m
7	1.87 m, 0.97 m	1.80 m, 1.13 m	1.81 m, 0.90 m
8	1.89 m	1.90 m	1.95 m
9	0.86 br d(10.8)	1.70 m	0.81 m
11	4.24 br s		4.29 br s
12	2.72 d(13.9)	2.51 d(12.0)	2.18 br d(14.0)
	1.68 m	2.25 m	1.40 m
14	1.48 m	1.67 m	0.88 m
15	2.09 m, 1.30 m	2.10 m, 1.40 m	2.02 m, 1.43 m
16	4.54 dt(8.0, 6.6)	4.49 dt(8.0, 7.5)	4.32 m
17	2.62 d(8.3)	2.12 d(8.0)	1.74 d(7.0)

Table 15-2-56 (continued)

H	15-2-173	15-2-174	15-2-175
18	5.33 s	1.05 s	1.39 s
19	1.09 s	1.01 s	1.07 s
21	1.43 s	1.29 s	1.31 s
23	2.10 m	2.02 dd(13.0, 7.0)	2.40 dd(12.0, 5.0)
23	1.75 m	1.77 t(13.0)	1.70 m
24	2.26 m	2.23 m	1.88 m
26	1.30 s	1.28 s	1.22 s
27	0.98 s	0.98 s	1.20 s
28	0.91 d(7.2)	0.94 d(7.0)	0.98 d(7.0)
OAc	2.09 s, 1.99 s		2.11 s
OH			3.19 s

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15.3 Marine alkaloids

Table 15-3-1: Compounds, MFs, and test solvents of marine alkaloids 15-3-1~15-3-11.

No.	Compounds	MFs	Test solvents	References
15-3-1	bauerine A	C ₁₂ H ₉ ClN ₂	CD ₃ COCD ₃	[81]
15-3-2	bauerine B	C ₁₂ H ₈ Cl ₂ N ₂	CD ₃ COCD ₃	[81]
15-3-3	bauerine C	C ₁₂ H ₈ Cl ₂ N ₂ O	DMSO- <i>d</i> ₆	[81]
15-3-4	nostocarboline	C ₁₂ H ₁₀ ClN ₂	CD ₃ OD	[82]
15-3-5	2-methyleudistomin D	C ₁₂ H ₁₀ BrN ₂ O	DMSO- <i>d</i> ₆	[83]
15-3-6	2-methyleudistomin J	C ₁₂ H ₁₀ BrN ₂ O	DMSO- <i>d</i> ₆	[83]
15-3-7	plakortamine B	C ₁₃ H ₉ BrN ₂	CDCl ₃	[84]
15-3-8	shepherdine	C ₁₂ H ₁₄ N ₂ O	CD ₃ OD	[85]
15-3-9	hyrtioerectine B	C ₁₃ H ₁₄ N ₂ O ₃	DMSO- <i>d</i> ₆	[86]
15-3-10	2-methyl-9 <i>H</i> -pyrido[3,4 <i>b</i>]indole-3-carboxylic acid	C ₁₃ H ₁₀ N ₂ O ₂	CD ₃ OD	[87]
15-3-11	8-hydroxy-2-methyl- β -carboline	C ₁₂ H ₁₁ N ₂ O	CD ₃ OD DMSO- <i>d</i> ₆	[88] [88]

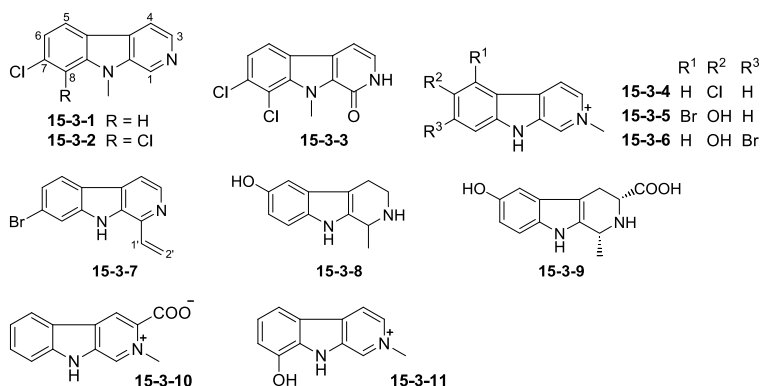


Table 15-3-2: ¹H NMR spectroscopic data of marine alkaloids 15-3-1~15-3-5.

H	15-3-1	15-3-2	15-3-3	15-3-4	15-3-5
1	9.02 s	9.04 s		9.23 br s	9.38 s
3	8.43 br d(4.8)	8.46 br d(5.4)	7.15 d(6.7)	8.52 dd(6.4, 0.9)	8.58 d(6.5)
4	8.07 d(4.8)	8.05 dd(5.4, 1.0)	7.02 d(6.7)	8.68 d(6.4)	8.96 d(6.5)
5	8.25 d(8.5)	8.18 d(8.3)	8.09 d(8.8)	8.49 dd(2.0, 0.8)	
6	7.28 dd(8.5, 1.6)	7.42 d(8.3)	7.44 d(8.8)		10.45 s(OH)
7				7.80 dd(8.9, 2.0)	7.53 d(9.0)
8	7.72 d(1.6)			7.77 dd(8.9, 0.8)	7.72 d(9.0)
NH			11.61 br s		12.99 s
NMe	4.04 s	4.38 s	4.63 s	4.53 s	4.49 s

Table 15-3-3: ^1H NMR spectroscopic data of marine alkaloids **15-3-6~15-3-9**.

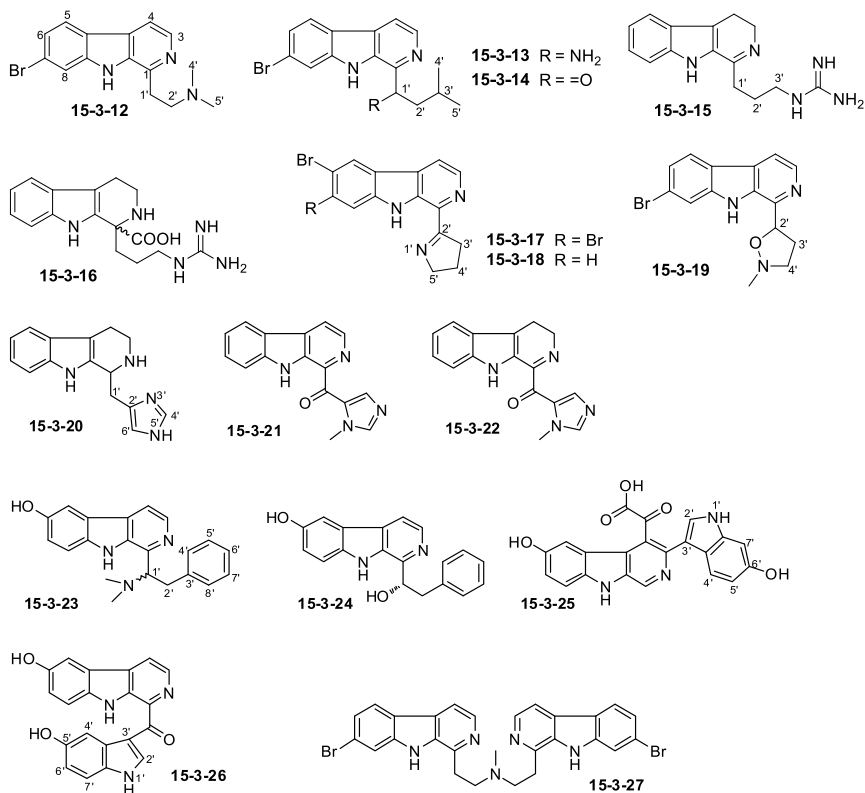
H	15-3-6	15-3-7	15-3-8	15-3-9
1	9.28 s		4.79 q(8)	4.52 m
3	8.54 d(6.5)	8.48 d(5.5)	3.45 m, 3.70 m	3.56 m
4	8.73 d(6.5)	7.84 d(5.5)	3.02 m	3.03 dd(15.5, 3.7) 2.68 dd(15.5, 14.3)
5	7.87 s	7.97 d(8)	6.84 d(2)	6.72 d(1.5)
6	10.53 s(OH)	7.41 dd(8, 1.5)		8.78 s(OH)
7			6.71 dd(2, 9)	6.58 dd(8.6, 1.5)
8	8.02 s	7.69 d(1.5)	7.18 d(9)	7.10 d(8.6)
1'		7.18 dd(17, 11)	1.72 d(8)	1.56 d(6.8)
2'		6.40 d(17), 5.72 d(11)		
NH	12.67 s	8.61 br s		10.70 s
NMe	4.44 s			

Table 15-3-4: ^1H NMR spectroscopic data of marine alkaloids **15-3-10** and **15-3-11**.

H	15-3-10	15-3-11 (CD_3OD)	15-3-11 ($\text{DMSO}-d_6$)
1	8.90 s	9.07 s	9.18 s
3		8.43 d(5.0)	8.58 d(6.0)
4	8.75 s	8.59 d(5.0)	8.72 d(6.0)
5	8.33 d(8.0)	7.84 d(8.0)	7.89 d(8.0)
6	7.45 dt(8.0, 8.0, 1.1)	7.27 t(8.0)	7.25 t(8.0)
7	7.77 m	7.15 d(8.0)	7.17 d(8.0)
8	7.70 m		10.70 s(OH)
NH			12.83 br s
NMe	4.62 s	4.51 s	4.51 s

Table 15-3-5: Compounds, MFs, and test solvents of marine alkaloids **15-3-12~15-3-27**.

No.	Compounds	MFs	Test solvents	References
15-3-12	plakortamine A	$\text{C}_{15}\text{H}_{16}\text{BrN}_3$	CDCl_3	[84]
15-3-13	eudistalbin A	$\text{C}_{16}\text{H}_{18}\text{BrN}_3$	CD_3CN	[89]
15-3-14	eudistalbin B	$\text{C}_{16}\text{H}_{14}\text{BrN}_2\text{O}$	CD_3CN	[89]
15-3-15	trypargimine	$\text{C}_{15}\text{H}_{19}\text{N}_5$	$\text{DMSO}-d_6$	[90]
15-3-16	1-carboxytrypargine	$\text{C}_{16}\text{H}_{22}\text{N}_5\text{O}_2$	$\text{DMSO}-d_6$	[90]
15-3-17	eudistomin V	$\text{C}_{15}\text{H}_{11}\text{Br}_2\text{N}_3$	CD_2Cl_2	[91]
15-3-18	eudistomin H	$\text{C}_{15}\text{H}_{12}\text{BrN}_3$	CD_2Cl_2	[91]
15-3-19	plakortamine D	$\text{C}_{15}\text{H}_{14}\text{BrN}_3\text{O}$	CDCl_3	[84]
15-3-20	haploscleridamine	$\text{C}_{15}\text{H}_{17}\text{N}_4$	CD_3OD	[92]
15-3-21	xestomanzamine A	$\text{C}_{16}\text{H}_{12}\text{N}_4\text{O}$	CDCl_3	[93]
15-3-22	xestomanzamine B	$\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}$	CDCl_3	[93]
15-3-23	eudistomin X	$\text{C}_{21}\text{H}_{21}\text{N}_3\text{O}$	CD_3OD	[94]
15-3-24	eudistomin W	$\text{C}_{19}\text{H}_{16}\text{N}_2\text{O}_2$	CD_3OD	[94]
15-3-25	hyrtioerectine A	$\text{C}_{21}\text{H}_{13}\text{N}_3\text{O}_5$	CD_3OD	[86]
15-3-26	hyrtiosulawesine	$\text{C}_{20}\text{H}_{13}\text{N}_3\text{O}_3$	CD_3OD	[85]
15-3-27	plakortamine C	$\text{C}_{27}\text{H}_{23}\text{Br}_2\text{N}_5$	CD_3OD	[84]

**Table 15-3-6:** ¹H NMR spectroscopic data of marine alkaloids 15-3-12~15-3-16.

H	15-3-12	15-3-13	15-3-14	15-3-15	15-3-16
3	8.28 d(5.5)	8.25 d(5.0)	8.53 d(5.0)	3.92 t(8.9)	3.38 m
4	7.78 d(5.5)	7.79 d(5.0)	8.10 d(5.0)	3.20 m	2.83 t(5.7)
5	7.95 d(8)	7.98 d(8.4)	8.00 d(8.4)	7.77 d(8.0)	7.38 d(7.8)
6	7.34 dd(8, 1.5)	7.31 dd(8.4, 1.3)	7.43 dd(8.4, 1.4)	7.19 t(7.4)	6.94 t(7.0)
7				7.45 t(7.5)	7.01 t(7.1)
8	7.67 d(1.5)	7.70 d(1.3)	7.75 d(1.5)	7.55 d(8.3)	7.30 d(8.0)
1'	3.36 t(5.5)	4.53 t(7.0)		3.04 t(7.7)	2.30 m, 1.98 m
2'	2.82 t(5.5)	1.72 m	3.30 d(7.0)	1.90 p(7.3)	1.60 m
3'		1.63 m	2.40 m	3.25 m	3.10 m
4'	2.47 s	0.88 d(7.0)	1.06 d(7.0)	7.95 t(5.3)	8.51 m
5'	2.47 s	0.94 d(7.0)	1.06 d(7.0)		
NH	12.52 br s			10.91 s	10.86 s

Table 15-3-7: ^1H NMR spectroscopic data of marine alkaloids **15-3-17~15-3-20**.

H	15-3-17	15-3-18	15-3-19	15-3-20
1				4.48 m
3	8.50 d(5.1)	8.47 d(5.1)	8.29 d(5.5)	3.36~3.32 m 3.04~3.01 m
4	7.95 d(5.1)	7.96 d(5.1)	7.95 d(5.5)	2.82~2.78 m
5	8.43 s	8.28 d(2.0)	7.99 d(8)	7.39 ddd(1.0, 1.0, 7.7)
6			7.41 dd(8, 1.5)	6.98 ddd(1.0, 7.7, 8.0)
7		7.64 dd(8.7, 2.0)		7.07 ddd(1.0, 8.0, 8.0)
8	7.93 s	7.49 d(8.7)	7.78 d(1.5)	7.31 ddd(1.0, 1.0, 8.0)
1'				3.36~3.32 m 3.04~3.01 m
2'			5.90 dd(8.5, 7)	
3'	3.27 ddt(9.1, 7.4, 2.1)	3.27 ddt(9.1, 7.5, 2.0)	3.08 m, 2.53 m	
4'	2.08 dddd (9.1, 7.5, 7.4, 7.3)	2.07 dddd (9.1, 7.5, 7.2, 7.5)	3.61 t(8) 2.80 m	7.65 d(1.2)
5'	4.25 ddt(7.5, 7.3, 2.1)	4.25 ddt(7.2, 7.5, 2.0)		
6				6.87 dt(1.2, 1.0)
NH	10.97 br s	10.93 br s	11.37 br s	
NMe			2.99 s	

Table 15-3-8: ^1H NMR spectroscopic data of marine alkaloids **15-3-21~15-3-24**.

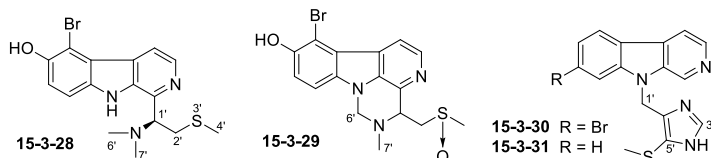
H	15-3-21	15-3-22	15-3-23	15-3-24
3	8.55 d(5.0)	4.16 dd(9.0, 9.0)	8.46 d(5.4)	8.10 d(6.0)
4	8.09 d(5.0)	2.97 dd(9.0, 9.0)	8.02 d(5.4)	8.39 d(6.0)
5	8.12 d(8.2)	7.60 d(7.9)	7.50 d(2.5)	7.64 d(2.2)
6	7.30 dd(8.2, 6.2)	7.13 dd(7.9, 7.0)		
7	7.55 dd(6.2, 7.3)	7.29 dd(7.0, 7.3)	7.13 dd(8.8, 2.5)	7.35 dd(8.8, 2.2)
8	7.57 d(7.3)	7.40 d(7.3)	7.34 d(8.8)	7.62 d(8.8)
1'			5.06 dd(5.0, 10.0)	5.77 t(5.8)
2'			3.65 m	3.40 (ov)
4'	8.93 s	8.37 s	6.95~7.01 m	6.98~7.12 m
5'			6.95~7.01 m	6.98~7.12 m
6'	7.66 s	7.63 s	6.95~7.01 m	6.98~7.12 m
7', 8'			6.95~7.01 m	6.98~7.12 m
NMe	4.05 s	3.99 s	3.00 br s	

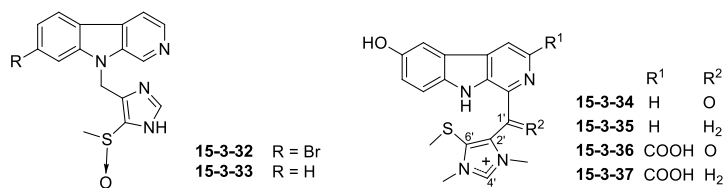
Table 15-3-9: ^1H NMR spectroscopic data of marine alkaloids **15-3-25**~**15-3-27**.

H	15-3-25	15-3-26	15-3-27 ^②
1	9.64 s		
3		8.41 d(4.8)	8.14 d(5.5)
4	12.44 s(COOH) ^①	8.14 d(4.8)	7.77 d(5.5)
5	8.07 d(2.0)	7.56 d(2.4)	7.96 d(8)
6	9.60 br s(OH) ^①		7.32 dd(8, 1.5)
7	6.80 dd(8.7, 2.0)	7.13 dd(2.4, 8.5)	
8	7.30 d(8.7)	7.53 d(8.5)	7.53 d(1.5)
1'	11.73 s ^①		3.31 t(5.5)
2'	8.87 s	8.88 s	3.09 t(5.5)
4'	7.57 d(8.7)	8.01 d(2.4)	
5'	7.13 dd(8.7, 2.2)		
6'	9.00 br s(OH) ^①	6.81 dd(2.4, 8.5)	
7'	7.60 d(2.0)	7.31 d(8.5)	
NMe			2.58 s

^① Recorded data in DMSO- d_6 .^② The compound **15-3-27** has a symmetrical structure and the ^1H NMR data of the symmetrical part overlaps.**Table 15-3-10:** Compounds, MFs, and test solvents of marine alkaloids **15-3-28**~**15-3-37**.

No.	Compounds	MFs	Test solvents	References
15-3-28	14-methyleudistomidin C	$\text{C}_{16}\text{H}_{18}\text{BrN}_3\text{OS}$	CD_3OD $\text{DMSO}-d_6$	[83] [83]
15-3-29	eudistomidin E	$\text{C}_{16}\text{H}_{16}\text{BrN}_3\text{O}_2\text{S}$	CDCl_3	[95]
15-3-30	didemnoline A	$\text{C}_{16}\text{H}_{13}\text{BrN}_4\text{S}$	$\text{DMSO}-d_6$ - CF_3COOD (95:5)	[96]
15-3-31	didemnoline B	$\text{C}_{16}\text{H}_{14}\text{N}_4\text{S}$	$\text{DMSO}-d_6$	[96]
15-3-32	didemnoline C	$\text{C}_{16}\text{H}_{13}\text{BrN}_4\text{OS}$	$\text{DMSO}-d_6$ - CF_3COOD (95:5)	[96]
15-3-33	didemnoline D	$\text{C}_{16}\text{H}_{14}\text{N}_4\text{OS}$	$\text{DMSO}-d_6$	[96]
15-3-34	hyrtiomanzamine	$\text{C}_{18}\text{H}_{17}\text{N}_4\text{O}_2\text{S}$	CD_3OD $\text{DMSO}-d_6$	[97] [97]
15-3-35	gesashidine A	$\text{C}_{18}\text{H}_{19}\text{N}_4\text{OS}$	$\text{DMSO}-d_6$	[98]
15-3-36	dragmacidonamine A	$\text{C}_{19}\text{H}_{17}\text{N}_4\text{O}_4\text{S}$	$\text{DMSO}-d_6$	[99]
15-3-37	dragmacidonamine B	$\text{C}_{19}\text{H}_{19}\text{N}_4\text{O}_3\text{S}$	$\text{DMSO}-d_6$	[99]



**Table 15-3-11:** ¹H NMR spectroscopic data of marine alkaloids **15-3-28** and **15-3-29**.

H	15-3-28 (CD ₃ OD)	15-3-28 (DMSO- <i>d</i> ₆)	15-3-29
3	8.47 d(5.5)	8.46 d(5.5)	8.39 d(5.5)
4	8.71 d(5.5)	8.57 d(5.5)	8.26 d(5.5)
6		10.03 brs(OH)	
7	7.28 d(8.5)	7.34 d(8.5)	7.35 d(8.7)
8	7.49 d(8.5)	7.55 d(8.5)	7.34 d(8.7)
1'	5.08 dd(8.0, 5.5)	5.31 dd(8.0, 5.5)	4.55 dd(10.5, 5.6)
2'	3.46 dd(14.0, 8.0)	3.40 m	3.42 dd(13.8, 5.6)
	3.47 dd(14.0, 5.5)		3.25 dd(13.8, 10.4)
4'	1.82 s	1.85 s	2.76 s
6'	2.93 s	2.82 s	5.32 d(12.4)
			5.15 d(12.4)
7'	2.93 s	2.82 s	2.50 s
NH		12.23 br s	

Table 15-3-12: ¹H NMR spectroscopic data of marine alkaloids **15-3-30~15-3-33**.

H	15-3-30	15-3-31	15-3-32	15-3-33
1	9.63 s	9.1 s	9.59 s	9.07 s
3	8.37 d(6.0)	7.91 d(5.1)	8.70 d(5.8)	8.13 d(5.2)
4	8.86 d(6.0)	8.33 d(5.1)	8.86 d(5.8)	8.39 d(5.2)
5	8.52 d(8.5)	8.11 d(8.0)	8.51 d(8.5)	8.26 d(8.2)
6	7.66 dd(8.5, 1.5)	7.60 dt(8.3, 1.1)	7.66 dd(8.5, 1.5)	7.58 t(7.5)
7		7.29 dt(8.3, 1.1)		7.29 t(7.5)
8	8.17 d(1.5)	7.80 d(8.0)	8.27 d(1.5)	7.75 d(8.0)
1'	6.03 s	5.57 s	6.10 s	5.85 s
4'	9.10 s	9.14 s	8.23 s	8.1 s
SMe	2.32 s	2.16 s	2.88 s	2.78 s

Table 15-3-13: ^1H NMR spectroscopic data of marine alkaloids **15-3-34**~**15-3-37**.

H	15-3-34 (DMSO- d_6)	15-3-34 (CD $_3$ OD)	15-3-35	15-3-36	15-3-37
3	8.46 s	8.46 d(4.9)	8.80 s		
4	8.46 s	8.35 d(4.9)	8.80 s	9.15 br s	8.69 br s
5	7.62 d(2.3)	7.61 d(2.4)	7.62 s	7.78 br d(1.9)	7.61 br d(2.2)
6	7.60 br s(OH)				
7	7.17 dd(2.3, 9)	7.20 dd(2.4, 8.8)	7.21 d(7.6)	7.20 dd(1.9, 8.2)	7.10 dd(2.2, 9.5)
8	7.63 d(8.3)	7.60 d(9)	7.56 d(7.6)	7.69 d(8.2)	7.69 d(9.5)
1'			4.86 s		4.80 s
4'	9.54 s		9.33 s	9.55 s	9.30 s
NH	12.05 s		12.19 s	12.40 br s	12.40 br s
NMe	3.90 s, 3.95 s	3.96 s, 4.03 s	3.84 s, 3.82 s	4.10 s, 3.91 s	3.87 s, 3.97 s
SMe	2.41 s	2.45 s	2.04 s	2.50 s	2.24 s

Table 15-3-14: Compounds, MFs, and test solvents of marine alkaloids **15-3-38**~**15-3-60**.

No.	Compounds	MFs	Test solvents	References
15-3-38	axiphenylalaninium	C $_{15}$ H $_{21}$ NO $_2$	CD $_3$ OD CDCl $_3$	[100] [100]
15-3-39	axityrosinium	C $_{15}$ H $_{21}$ NO $_3$	CD $_3$ OD	[100]
15-3-40	1-carboxy-6-hydroxy-3,4-dihydro- β -carboline	C $_{12}$ H $_{10}$ N $_2$ O $_3$	DMSO- d_6	[101]
15-3-41	pyrodysinoic acid	C $_{17}$ H $_{23}$ NO $_3$	DMSO- d_6	[102]
15-3-42	isopyrodysinoic acid	C $_{17}$ H $_{23}$ NO $_3$	DMSO- d_6	[102]
15-3-43	13-hydroxyisopyrodysinoic acid	C $_{17}$ H $_{23}$ NO $_4$	DMSO- d_6	[102]
15-3-44	pyrodysinoic acid B	C $_{17}$ H $_{23}$ NO $_3$	DMSO- d_6	[102]
15-3-45	antipathine A	C $_{16}$ H $_{13}$ N $_3$ O $_2$	C $_5$ D $_5$ N	[103]
15-3-46	(+)-dihydrodiscorhabdin A	C $_{18}$ H $_{17}$ BrN $_3$ O $_2$ S	CD $_3$ OD	[104]
15-3-47	(+)-discorhabdin X	C $_{18}$ H $_{18}$ N $_3$ O $_3$ S	CD $_3$ OD	[104]
15-3-48	saldedine A	C $_{18}$ H $_{17}$ Br $_2$ NO $_3$	CDCl $_3$	[105]
15-3-49	<i>N, N</i> -dimethylsaldedine A	C $_{19}$ H $_{20}$ Br $_2$ NO $_3$	DMSO- d_6	[105]
15-3-50	saldedine B	C $_{18}$ H $_{19}$ Br $_2$ NO $_3$	CDCl $_3$	[105]
15-3-51	clavatadine C	C $_{14}$ H $_{17}$ Br $_2$ N $_5$ O $_3$	DMSO- d_6	[106]
15-3-52	clavatadine D	C $_{15}$ H $_{19}$ Br $_2$ N $_5$ O $_3$	DMSO- d_6	[106]
15-3-53	clavatadine E	C $_{14}$ H $_{20}$ BrN $_5$ O $_3$	DMSO- d_6	[106]
15-3-54	mukanadin E	C $_{12}$ H $_{19}$ Br $_2$ N $_5$ O	DMSO- d_6	[107]
15-3-55	mukanadin F	C $_{11}$ H $_{10}$ Br $_2$ N $_4$ O $_4$	DMSO- d_6	[107]
15-3-56	aphrocallistin	C $_{20}$ H $_{24}$ Br $_2$ N $_6$ O $_2$	CD $_3$ OD	[108]
15-3-57	leptoclinidamine A	C $_{16}$ H $_{19}$ N $_5$ O $_4$	DMSO- d_6	[109]
15-3-58	leptoclinidamine B	C $_{16}$ H $_{19}$ N $_5$ O $_5$	DMSO- d_6	[109]
15-3-59	leptoclinidamine C	C $_{18}$ H $_{20}$ BrN $_4$ O $_3$ S	DMSO- d_6	[109]
15-3-60	19-hydroxyaraplypsillin-I <i>N</i> ²⁰ -sulfonate sodium	C $_{21}$ H $_{21}$ Br $_4$ N $_3$ NaO $_9$ S	CD $_3$ CN, DMSO- d_6	[110]

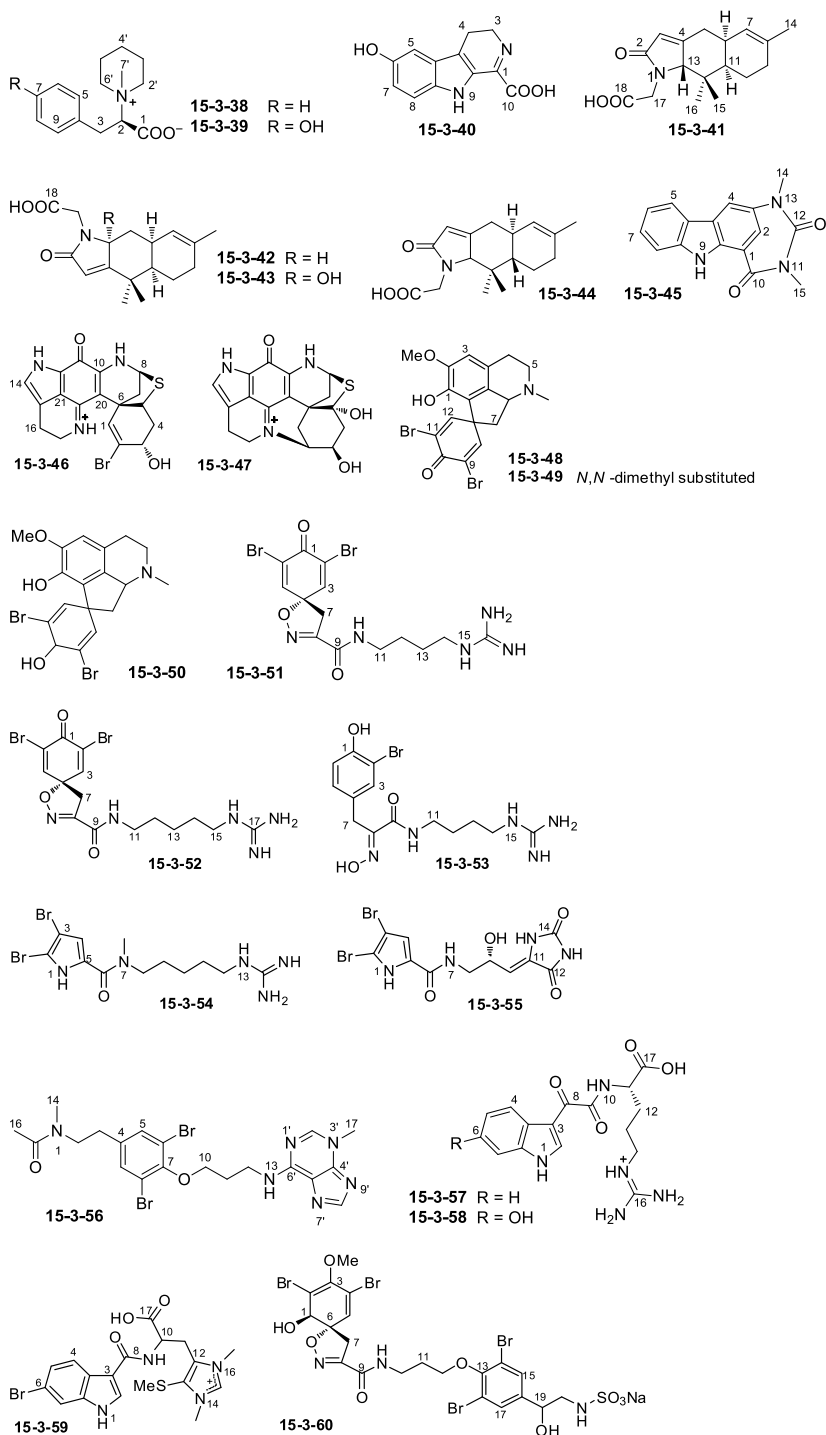


Table 15-3-15: ^1H NMR spectroscopic data of marine alkaloids **15-3-38~15-3-40**.

H	15-3-38 (CD_3OD)	15-3-38 (CDCl_3)	15-3-39	15-3-40
2	3.89 m	4.11 dd(5.6, 8.0)	3.87 dd(4.0, 10.6)	
3	3.20 m	3.57 dd(8.0, 13.8)	3.17 dd(4.0, 12.6)	3.83 t(8.1)
	3.17 m	3.13 dd(5.6, 13.8)	3.13 dd(10.6, 12.6)	
4				3.08 t(8.1)
5	7.27 m	7.31~7.35 m	7.12 d(7.3)	6.86 br s
6	7.27 m	7.31~7.35 m	6.72 d(7.3)	
7	7.19 m	7.25 m		6.96 br d(8.8)
8	7.27 m	7.31~7.35 m	6.72 d(7.3)	7.47 d(8.8)
9	7.27 m	7.31~7.35 m	7.12 d(7.3)	
10				11.65 br s(COOH)
2'	3.66 m	4.00 ddd(3.2, 9.2, 13.6)	3.68 ddd(3.9, 9.0, 12.7)	
	3.36 m	3.37 m	3.37 m	
3'	1.86~1.99 m	2.10 m, 1.84 m	1.88~2.00 m	
4'	1.75 m, 1.68 m	1.69 m, 1.66 m	1.74 m, 1.67 m	
5'	1.86~1.99 m	1.86 m, 1.78 m	1.88~2.00 m	
6'	3.56 m, 3.52 m	3.30 m	3.56 m, 3.52 m	
7'	3.28 s	3.35 s	3.29 s	

Table 15-3-16: ^1H NMR spectroscopic data of marine alkaloids **15-3-41~15-3-44**.

H	15-3-41	15-3-42	15-3-43	15-3-44
3	5.78 s	5.67 s	5.66 s	5.82 s
4		4.17 dd(6.1, 12.1)	5.93 br s(OH)	
5ax	2.14 dd(13.8, 13.8)	0.87 ddd(12.1, 12.1, 12.1)	1.29 dd(13.5, 13.5)	1.94 dd(11.9, 11.9)
5eq	2.71 dd(4.9, 13.8)	2.25 m	1.93 dd(3.6, 13.5)	2.72 dd(1.7, 11.9)
6	2.36 m	2.59 m	2.67 m	1.97 m
7	5.41 br d(4.3)	5.36 br d(4.8)	5.34 br d(5.1)	5.17 br s
9	1.98 m	ax 1.86 m, eq 1.91 m	1.90 m	1.97 m
10ax	1.58 m	1.03 ddd(13.5, 13.5, 6.2)	1.02 m	1.15 m
10eq	1.76 br d(11.5)	1.59 m	1.58 m	1.86 br d(10.1)
11	1.38 br d(11.0)	1.59 m	1.55 m	1.18 br t(11.3)
13	3.97 br s			3.88 br s
14	1.61 br s	1.57 br s	1.56 br s	1.62 br s
15	0.70 s	1.21 s	1.32 s	1.14 s
16	1.18 s	1.16 s	1.13 s	0.51 s
17	3.81 d(18.0), 4.27 d(18.0)	3.85 d(17.8), 4.07 d(17.8)	3.72 d(17.8), 3.89 d(17.8)	3.87 d(18.0), 4.39 d(18.0)
COOH	12.72 br s	12.67 br s	12.54 br s	12.78 br s

Table 15-3-17: ^1H NMR spectroscopic data of marine alkaloids 15-3-45~15-3-48.

H	15-3-45	15-3-46	15-3-47	15-3-48
1		6.47 s	2.56 dd(13.1, 2.5) 2.02 dd(13.1, 3.3)	
2	8.66 s		4.10 m(ov)	
3		4.32 dd(2.8, 2.8)	4.29 m(ov)	6.60 s
4	8.14 s	2.17 ddd(2.8, 13.6, 13.6) 2.00 ddd(2.8, 4.9, 13.6)	2.37 dd(13.1, 5.1) 2.24 dd(13.1, 11.1)	2.90 m 2.78 dd(16.7, 4.7)
5	7.66 d(7.5)	4.34 dd(13.6, 4.9)		3.12 dd(11.8, 6.7) 2.50 m
6	7.35 t(7.5)			
6a				3.45 dd(9.5, 6.1)
7	7.60 t(7.5)	2.60 dd(12.3, 3.6) 2.46 br d(12.3)	2.87 dd(11.9, 4.0) 2.05 d(11.9)	2.50 m 2.28 m
8	8.42 d(7.5)	5.20 dd(3.6, 1.1)	5.29 d(4.0)	7.42 d(2.2)
12				7.30 d(2.2)
13				2.39 s
14	3.66 s	7.17 s	7.11 s	3.83 s
15	3.57 s			
16		2.96 m	3.12 m, 3.05 m	
17		3.95 m, 3.87 m	4.24 m(ov), 4.07 m(ov)	
NH	12.73 s			

Table 15-3-18: ^1H NMR spectroscopic data of marine alkaloids 15-3-49 and 15-3-50.

H	15-3-49	15-3-50	H	15-3-49	15-3-50
1	9.19 s(OH)		8	7.61 d(2.3)	6.28 s
3	6.92 s	6.65 s	10		4.56
4	3.07 m	3.00 m 2.76 dd(16.8, 4.9)	12	7.82 d(2.3)	6.16 s
5	3.89 dd(9.7, 6.9) 3.63 m	3.21 m 2.52 m	13	3.19 s 2.80 s	2.44 s
6a	5.33 dd(9.7, 6.9)	3.48 m	14	3.77 s	3.82 m
7	2.70 m, 2.60 m	2.46 m, 2.09 m			

Table 15-3-19: ^1H NMR spectroscopic data of marine alkaloids 15-3-51~15-3-56.

H	15-3-51	15-3-52	15-3-53	15-3-54	15-3-55	15-3-56
1	7.80 s	7.79 s	10.02 s(OH)	12.27 br s	12.67 br s	
2						3.50 t(7.6)
3			7.28 d(1.8)			2.74 t(7.6)
4				6.60 br s	6.93 br s	

Table 15-3-19 (continued)

H	15-3-51	15-3-52	15-3-53	15-3-54	15-3-55	15-3-56
5	7.80 s	7.79 s	7.01 dd(8.4, 1.8)			7.45 s
6			6.83 d(8.4)			
7	3.55 s	3.55 s	3.69 s		8.06 t(5.4)	
8				3.44 t	3.29 m	
9				1.58 m	4.43 m	7.45 s
					5.34 m(OH)	
10	8.63 t	8.59 t	7.99 t(6.0)	1.30 m	5.45 d(8.4)	4.11 m
11	3.18 dt(6.0, 6.0)	3.16 dt(6.0, 6.0)	3.15 dt(6.0, 6.0)	1.52 m		2.27 m
12	1.49 m	1.49 tt(6.0, 6.0)	1.43 m	3.12		4.04 m
13	1.48 m	1.29 tt(6.0, 6.0)	1.42 m	7.02	10.02 br s	
14	3.11 dt(6.0, 6.0)	1.48 tt(6.0, 6.0)	3.08 dt(6.0, 6.0)			2.98 s
15	7.45 m	3.08 dt(6.0, 6.0)	7.39 t(6.0)		11.02 br s	
16		7.41 br t(6.0)				2.02 s
17				3.08 s		4.06 s
2'						8.64 s
8'						8.42 s
NOH			11.75 s			

Table 15-3-20: ¹H NMR spectroscopic data of marine alkaloids 15-3-57~15-3-60.

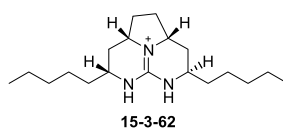
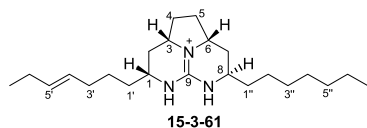
H	15-3-57	15-3-58	15-3-59	15-3-60 (CD ₃ CN)	15-3-60 (DMSO- <i>d</i> ₆)
1	12.32 br s	11.86 br s	11.74 br s	4.15 m	3.92 dd(8.1, 0.9) 6.38 d(8.1, OH)
2	8.67 d(3.0)	8.63 d(2.9)	8.08 d(2.9)		
3				3.72 s(OMe)	3.64 s(OMe)
4	8.21 dd(2.4, 6.0)	7.97 d(7.9)	7.96 d(7.9)		
5	7.26 m	6.73 dd(1.8, 7.9)	7.24 dd(1.8, 7.9)	6.43 s	6.59 d(0.9)
6	7.26 m	9.29 s(OH)			
7	7.54 dd(1.8, 6.0)	6.86 d(1.8)	7.65 d(1.8)	3.71 d(18.3) 3.08 d(18.3)	3.62 d(18.4) 3.22 d(18.4)
9			8.42 d(7.8)		8.58 t(5.8, NH)
10	8.83 d(7.8)	8.42 br d(7.8)	4.87 ddd(5.6, 7.8, 9.8)	3.55 t(6.8)	3.40 m
11	4.32 ddd(4.8, 7.8, 7.8)	4.05 m	3.27 dd(9.8, 15.6) 3.43 dd(5.6, 15.6)	2.12 m	1.99 p(6.8)
12	1.78 m, 1.88 m	1.74 m, 1.79 m		4.07 t(6.2)	3.96 t(6.6)
13	1.54 tt(7.2, 7.2)	1.51 tt(7.2, 7.2)	2.31 s(SMe)		

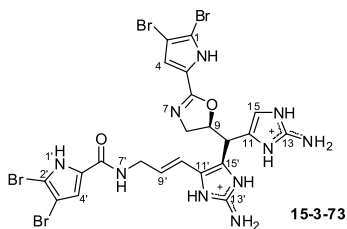
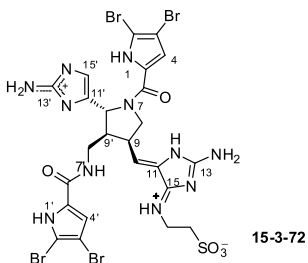
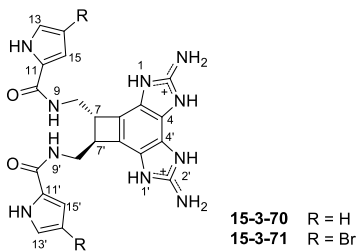
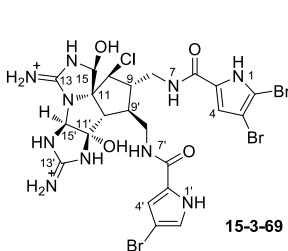
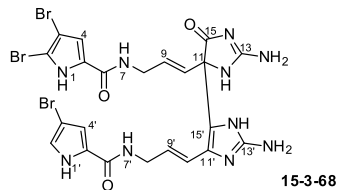
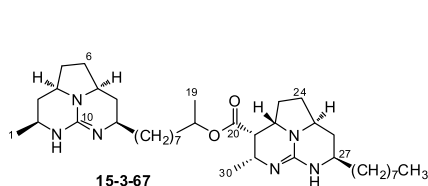
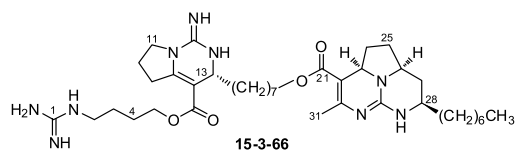
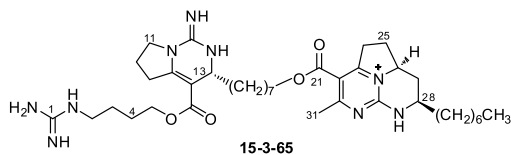
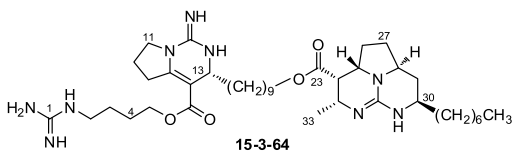
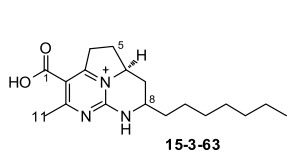
Table 15-3-20 (continued)

H	15-3-57	15-3-58	15-3-59	15-3-60 (CD ₃ CN)	15-3-60 (DMSO- <i>d</i> ₆)
14	3.12 dt(5.4, 7.2)	3.10 td(7.2, 7.8)	3.79 s(NMe)		
15	7.66 t(5.4)	8.10 br s	9.17 s	7.60 s	7.56 s
16	7.22 br s(N ₂ H ₄)	7.12 br s(N ₂ H ₄)	3.89 s(NMe)		
17				7.60 s	7.56 s
19				4.71 m	4.66 m
					5.76 d(4.3, OH)
20				3.20 dd(14.3, 2.9)	2.90 m
				3.07 dd(14.3, 2.9)	6.79 br(NH)

Table 15-3-21: Compounds, MFs, and test solvents of marine alkaloids 15-3-61~15-3-80.

No.	Compounds	MFs	Test solvents	References
15-3-61	merobatzelladine A	C ₂₃ H ₄₂ N ₃	CD ₃ OD	[111]
15-3-62	merobatzelladine B	C ₁₉ H ₃₆ N ₃	CD ₃ OD	[111]
15-3-63	clathriadic acid	C ₁₈ H ₂₈ N ₃ O ₂	CD ₃ OD	[112]
15-3-64	dinorbatzelladine A	C ₄₀ H ₆₉ N ₉ O ₄	CD ₃ OD	[112]
15-3-65	dinordehydrobatzelladine B	C ₃₈ H ₆₂ N ₉ O ₄	CD ₃ OD	[112]
15-3-66	dinorbatzelladine B	C ₃₈ H ₆₄ N ₉ O ₄	CD ₃ OD	[112]
15-3-67	norbatzelladine L	C ₃₈ H ₆₆ N ₆ O ₂	CD ₃ OD	[112]
15-3-68	nagelamide P	C ₂₂ H ₂₀ Br ₃ N ₁₀ O ₃	DMSO- <i>d</i> ₆	[107]
15-3-69	nagelamide O	C ₂₂ H ₂₄ Br ₃ ClN ₁₀ O ₄	DMSO- <i>d</i> ₆	[107]
15-3-70	benzosceptrin A	C ₂₂ H ₂₂ N ₁₀ O ₂	CD ₃ OD	[113]
15-3-71	benzosceptrin B	C ₂₂ H ₂₀ Br ₂ N ₁₀ O ₂	CD ₃ OD	[113]
15-3-72	nagelamide Q	C ₂₄ H ₂₆ Br ₄ N ₁₁ O ₅ S	DMSO- <i>d</i> ₆	[114]
15-3-73	nagelamide R	C ₂₂ H ₂₂ Br ₄ N ₁₀ O ₂	C ₅ D ₅ N	[114]
15-3-74	tyrokeradine A	C ₂₉ H ₃₅ Br ₂ N ₇ O ₇	DMSO- <i>d</i> ₆	[115]
15-3-75	tyrokeradine B	C ₂₆ H ₂₈ Br ₂ N ₇ O ₇	DMSO- <i>d</i> ₆	[115]
15-3-76	fasciospongine A	C ₃₀ H ₄₇ N ₃ O ₅ S	CD ₃ OD	[116]
15-3-77	fasciospongine B	C ₃₀ H ₄₇ N ₃ O ₅ S	CD ₃ OD	[116]
15-3-78	19-oxofasciospongine A	C ₃₀ H ₄₄ N ₃ NaO ₆ S	CD ₃ OD	[116]
15-3-79	fasciospongine C	C ₃₀ H ₅₂ N ₄ O ₅ S	CD ₃ OD	[116]
15-3-80	25-hydroxyhalisulfate 9	C ₂₅ H ₃₉ O ₇ S	CD ₃ OD	[116]





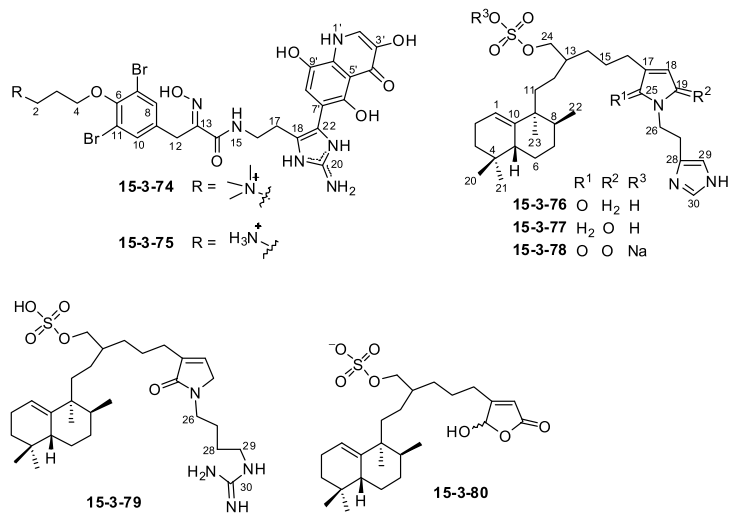


Table 15-3-22: ¹H NMR spectroscopic data of marine alkaloids **15-3-61** and **15-3-62**.

H	15-3-61	15-3-62	H	15-3-61	15-3-62
1	3.43 m	3.43 m	4'	5.32 m	1.35 m
2	1.25 m, 2.25 m	1.26 m, 2.27 m	5'	5.38 m	0.92 m
3	3.76 m	3.76 m	6'	2.04 q(7.0)	
4	1.70 m, 2.23 m	1.68 m, 2.23 m	7'	0.95 t(7.0)	
5	1.64 m, 2.24 m	1.66 m, 2.24 m	1''	1.48 m, 1.56 m	1.49 m, 1.57 m
6	3.73 m	3.74 m	2''	1.36 m	1.40 m
7	1.57 m, 2.16 m	1.58 m, 2.17 m	3''	1.30 m	1.35 m
8	3.49 m	3.49 m	4''	1.30 m	1.35 m
1'	1.54 m, 1.59 m	1.54 m, 1.59 m	5''	1.29 m	0.92 m
2'	1.45 m	1.40 m	6''	1.30 m	
3'	2.09 m	1.35 m	7''	0.89 t(7.0)	

Table 15-3-23: ¹H NMR spectroscopic data of marine alkaloid **15-3-63**.

H	15-3-63	H	15-3-63	H	15-3-63
4	3.66 dd(19.7, 8.9)	11	2.76 s	15	1.33 m
5	2.65 m	12	1.58 m	16	1.33 m
6	4.57 m	13	1.47 m	17	1.38 m
7	2.57 dt(13.5, 3.7)	14	1.33 m	18	0.91 t(7.0)
	1.57 m				
8	3.81 ddd(11.7, 8.3, 4.0)				

Table 15-3-24: ¹H NMR spectroscopic data of marine alkaloids **15-3-64**~**15-3-67**.

H	15-3-64	15-3-65	15-3-66	15-3-67
1				1.28 d(6.5)
2	3.23 t(6.9)	3.23 t(6.9)	3.23 t(6.8)	3.55 m
3	1.69 m	1.68 m	1.69 m	2.25 m, 1.26 m
4	1.77 m	1.76 m	1.77 m	3.75 m
5	4.22 t(6.3)	4.22 t(6.5)	4.22 t(6.5)	2.23 m, 1.69 m
6				2.21 m, 1.69 m
7				3.75 m
8				2.27 m, 1.24 m
9	3.32 m	3.32 m	3.32 m	3.43 m
	2.29 m	2.98 dt(18.0, 9.5)	2.98 dt(18.1, 9.8)	
10	2.23 m, 2.11 m	2.24 m, 2.11 m	2.23 m, 2.11 m	
11	3.82 m	3.81 dt(9.2, 2.1)	3.82 dt(9.2, 2.1)	1.57 m
	3.67 m	3.67 dt(17.0, 9.5)	3.68 dt(17.1, 9.5)	
12				1.34 m
13	4.40 t(6.1)	4.39 t(5.8)	4.40 t(6.3)	1.34 m
14	1.58 m	1.59 m	1.58 m	1.34 m
15	1.41 m	1.43 m	1.42 m	1.34 m
16	1.33 m	1.33 m	1.33 m	1.35 m
17	1.33 m	1.33 m	1.33 m	1.63 m, 1.56 m
18	1.33 m	1.38 m	1.33 m	4.98 m
19	1.33 m	1.78 m	1.65 m	1.26 d(6.2)
20	1.37 m	4.33 t(6.5)	4.14 m	
21	1.65 m			3.10 dd(4.5, 3.5)
22	4.14 t(6.5)			3.95 m
23			4.53 dd(9.2, 6.4)	2.26 m, 1.65 m
24	3.13 dd(4.7, 3.3)	3.60 d(8.4)	2.53 ddd(12.0, 7.4, 4.1)	2.20 m, 1.64 m
			1.67 m	
25	3.95 m	2.65 dd(12.5, 6.4)	2.10 m, 1.57 m	3.55 m
26	2.24 m	4.57 m	3.82 m	2.36 ddd(12.5, 4.7, 2.0)
	1.60 m			1.45 m
27	1.66 m, 2.22 m	2.58 dt(13.2, 3.5)	2.43 ddd(11.2, 6.7, 3.0)	3.54 m
		1.57 m	1.32 m	
28	3.55 m	3.81 m	3.47 m	
29	2.36 ddd(12.7, 4.9, 2.5)			3.85 m
	1.43 m			
30	3.54 m			1.26 d(6.2)
31		2.75 s	2.30 s	1.63 m, 1.56 m
32	3.85 m	1.58 m	1.58 m	1.34 m
33	1.27 d(6.7)	1.47 m	1.44 m	1.34 m
34	1.58 m	1.33 m	1.33 m	1.34 m
35	1.39 m	1.33 m	1.33 m	1.34 m
36	1.33 m	1.33 m	1.33 m	1.34 m
37	1.33 m	1.38 m	1.38 m	1.38 m

Table 15-3-24 (continued)

H	15-3-64	15-3-65	15-3-66	15-3-67
38	1.29 m	0.94 t(6.5)	0.90 t(7.0)	0.90 t(7.1)
39	1.29 m			
40	0.90 t(7.0)			

Table 15-3-25: ^1H NMR spectroscopic data of marine alkaloids 15-3-68~15-3-71.

H	15-3-68	15-3-69	15-3-70	15-3-71
1	12.68 br s	12.71 br s		
4	6.95 br s	7.00 br s		
7	8.42 brt(5.9)	8.34 brt(5.7)	3.69 dd(8.7, 4.3)	3.67 dd(8.6, 4.0)
8	3.87 brt(5.3)	3.24 m, 3.73 m	3.95 dd(13.4, 4.3) 3.54 dd(13.4, 8.7)	3.93 dd(11.0, 4.0) 3.53 dd(11.0)
9	6.02 dt(16.0, 5.3)	1.95 m		
10	6.05 br d	4.27 d(12.3)		
13	7.62 br s(NH ₂)	9.27 br s(NH ₂) 8.87 br s(NH ₂)	6.86 dd(2.6, 1.4)	6.86 dd(2.4)
14	12.12 br s	10.19 br s	6.08 dd(3.8, 2.6)	
15		5.28 br d(6.4) 7.53 br d(6.4, OH)	6.61 dd(3.8, 1.4)	6.61 dd(3.8)
1'	11.80 br s	11.82 br s		
2'	6.98 br s	6.90 br s		
4'	6.86 br s	6.98 br s		
7'	8.39 brt(5.9)	8.21 br s	3.69 dd(8.7, 4.3)	3.67 dd(8.6, 4.0)
8'	3.95 m	3.26 m, 3.44 m	3.95 dd(13.4, 4.3) 3.54 dd(13.4, 8.7)	3.93 dd(11.0, 4.0) 3.53 dd(11.0)
9'	6.02 dt(16, 5.3)	2.61 br s		
10'	6.41 br d(16)	2.87 br d(4)		
11'		7.82 s(OH)		
12'	12.56 br s	9.92 br s		
13'	7.62 br s(NH ₂)	8.62 br s(NH ₂)	6.86 dd(2.6, 1.4)	6.86 dd(2.4)
14'		9.25 br s	6.08 dd(3.8, 2.6)	
15'		5.32 br s	6.61 dd(3.8, 1.4)	6.61 dd(3.8)

Table 15-3-26: ^1H NMR spectroscopic data of marine alkaloids 15-3-72 and 15-3-73.

H	15-3-72	15-3-73	H	15-3-72	15-3-73
1	12.61 br s		7'	8.11 br t(5.2)	9.02 br t(5.7)
4	6.77 s	7.11 br s	8'	3.23 m	4.18 m
8	4.23 m 3.37 m	3.98 dd(15.0, 7.3) 4.23 m	9'	2.77 m	6.48 dt(15.9, 6.1)
			10'	4.90 d(6.3)	6.75 d(15.9)

Table 15-3-26 (continued)

H	15-3-72	15-3-73	H	15-3-72	15-3-73
9	3.48 m	5.50 m	12'	12.47 br s	
10	6.22 d(10.2)	4.73 d(5.9)	13'	7.44 br s(NH ₂)	
12	11.54 br s		14'	11.95 br s	
13	9.23 br s(NH ₂)		15'	6.79 s	
	8.69 br s(NH ₂)		1''	9.64 br t(5.6)	
15		7.17 s	2''	3.64 m	
1'	12.61 br s		3''	2.75 m	
4'	6.81 s	7.21 br s			

Table 15-3-27: ¹H NMR spectroscopic data of marine alkaloids 15-3-74 and 15-3-75.

H	15-3-74	15-3-75	H	15-3-74	15-3-75
1	3.11 s	7.88 br s	17	2.71 t(7.2)	2.71 t(7.1)
2	3.59 t(7.3)	3.04 br t(6.6)	19	12.05 br s	12.09 br s
3	2.22 m	2.05 m	20	7.30 s(NH ₂)	7.39 s(NH ₂)
4	3.96 t(5.4)	3.96 t(5.8)	21	12.28 br s	12.36 br s
8	7.42 s	7.41 s	1'	11.73 d(5.8)	11.73 d(5.8)
10	7.42 s	7.41 s	2'	7.66 d(5.8)	7.66 d(5.8)
12	3.66 s	3.66 s	3'	8.92 s(OH)	8.92 s(OH)
13	11.98 s(NOH)	11.98 s(NOH)	6'	14.32 s(OH)	14.28 s(OH)
15	8.10 br t(5.7)	8.07 br t(5.7)	8'	6.91 s	6.85 s
16	3.34 dt	3.36 dt	9'	10.30 s(OH)	10.30 s(OH)

Table 15-3-28: ¹H NMR spectroscopic data of marine alkaloids 15-3-76~15-3-80.

H	15-3-76	15-3-77	15-3-78	15-3-79	15-3-80
1	5.34 dd(2.3, 4.8)	5.34 dd(2.3, 4.8)	5.34 dd(2.5, 4.5)	5.34 dd(2.3, 4.8)	5.34 dd(2.3, 4.8)
2α	1.98 m	1.98 m	1.98 m	1.98 m	1.98 m
2β	2.07 m	2.07 m	2.07 m	2.07 m	2.07 m
3α	1.39 m	1.39 m	1.39 m	1.39 m	1.39 m
3β	1.09 m	1.09 m	1.09 m	1.09 m	1.09 m
5	1.64 dd(2.5, 13.0)	1.64 m	1.64 br d(13.0)	1.64 m	1.64 dd(2.5, 13.0)
6α	1.08 m	1.08 m	1.08 m	1.08 m	1.08 m
6β	1.83 m	1.84 m	1.83 m	1.83 m	1.84 m
7α	1.53 m	1.54 m	1.54 m	1.53 m	1.54 m
7β	1.51 m	1.52 m	1.52 m	1.51 m	1.52 m
8	1.25 m	1.25 m	1.25 m	1.25 m	1.25 m
11	1.75 m, 1.05 m	1.75 m, 1.05 m	1.75 m, 1.05 m	1.75 m, 1.05 m	1.75 m, 1.05 m

Table 15-3-28 (continued)

H	15-3-76	15-3-77	15-3-78	15-3-79	15-3-80
12	1.10 m, 1.04 m	1.10 m, 1.04 m	1.10 m, 1.04 m	1.10 m, 1.04 m	1.10 m, 1.05 m
13	1.59 m	1.59 m	1.59 m	1.58 m	1.62 m
14	1.44 m, 1.29 m	1.44 m, 1.31 m	1.44 m, 1.29 m	1.44 m, 1.29 m	1.52 m, 1.40 m
15	1.57 m	1.61 m	1.61 m	1.60 m	1.68 m
16a	2.24 dd(15.0, 7.0)	2.48 dd(15.0, 7.0)	2.43 ddd(15.0, 7.0, 1.5)	2.43 ddd(15.0, 7.0, 1.5)	2.44 m
16b	2.19 dd(15.0, 7.5)	2.42 dd(15.0, 7.5)	2.36 dd(15.0, 7.5, 1.5)	2.30 dd(15.0, 7.5, 1.5)	2.39 m
18	6.84 t(1.5)	5.76 s	6.41 t(1.5)	6.91 t(1.5)	5.90 s
19	3.89 d(1.5)			3.98 br s	
20	0.90 s	0.90 s	0.91 s	0.91 s	0.91 s
21	0.85 s	0.85 s	0.86 s	0.86 s	0.86 s
22	0.86 d(7.0)	0.86 d(7.0)	0.87 d(7.0)	0.87 d(7.0)	0.87 d(7.0)
23	1.01 s	1.02 s	1.02 s	1.02 s	1.02 s
24	3.94 dd(5.0, 9.5)	3.99 dd(4.0, 9.5)	3.95 dd(4.5, 9.5)	3.93 dd(5.0, 9.5)	3.97 dd(4.5, 9.5)
	3.81 dd(6.8, 9.5)	3.81 dd(7.0, 9.5)	3.80 dd(6.5, 9.5)	3.79 dd(7.0, 9.5)	3.84 dd(6.8, 9.5)
25		4.08 d(20.5)			6.02 br s
		4.02 d(20.5)			
26	3.77 dd(14.0, 7.0)	3.75 dd(13.8, 6.8)	3.78 t(6.5)	3.55 dd(14.0, 6.5)	
	3.72 dd(14.0, 6.5)	3.70 dd(13.8, 7.0)	3.78 t(6.5)	3.50 dd(14.0, 6.5)	
27	2.94 t(6.8)	3.02 t(6.8)	2.97 t(6.5)	1.68 m	
28				1.52 m	
29	7.00 br s	7.25 br s	7.16 br s	3.22 t(7.5)	
30	8.06 br s	8.55 br s	8.45 br s		

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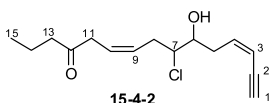
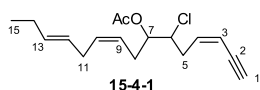
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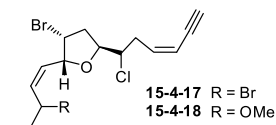
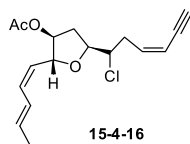
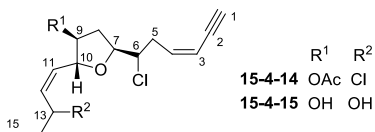
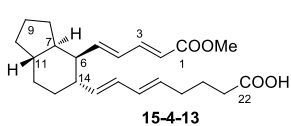
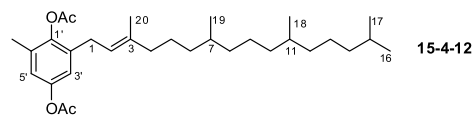
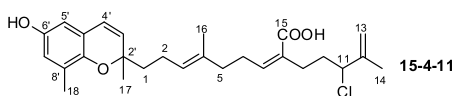
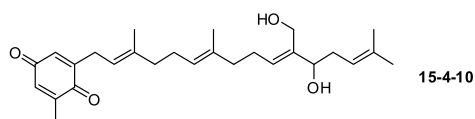
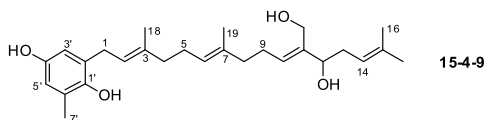
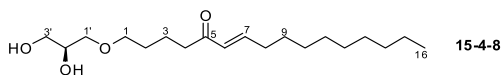
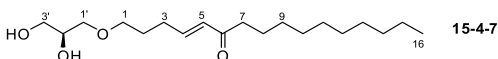
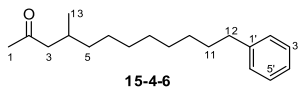
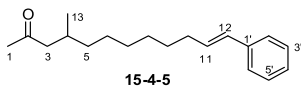
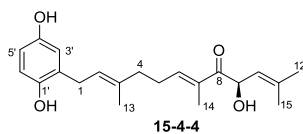
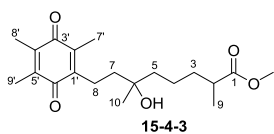
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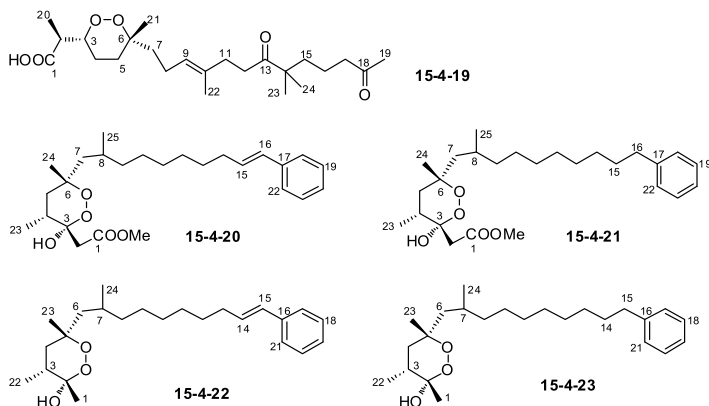
15.4 Some additional marine natural products

Table 15-4-1: Compounds, MFs, and test solvents of marine natural products **15-4-1~15-4-23**.

No.	Compounds	MFs	Test solvents	References
15-4-1	(3 <i>Z</i> ,9 <i>Z</i> ,12 <i>E</i>)-7-acetoxy-6-chloropentadeca-3,9,12-trien-1-yne	C ₁₇ H ₂₃ ClO ₂	CDCl ₃	[117]
15-4-2	(3 <i>Z</i> ,9 <i>Z</i>)-7-chloro-6-hydroxy-12-oxo-pentadeca-3,9-dien-1-yne	C ₁₅ H ₂₁ ClO ₂	CDCl ₃	[118]
15-4-3	sarcophytonone	C ₂₀ H ₃₀ O ₅	CDCl ₃	[119]
15-4-4	rossinone A	C ₂₁ H ₂₈ O ₄	CDCl ₃	[120]
15-4-5	—	C ₁₉ H ₂₈ O	CDCl ₃	[121]
15-4-6	—	C ₁₉ H ₃₀ O	CDCl ₃	[121]
15-4-7	ceratodictyol A	C ₁₉ H ₃₆ O ₄	CD ₃ OD	[122]
15-4-8	ceratodictyol B	C ₁₉ H ₃₆ O ₄	CD ₃ OD	[122]
15-4-9	fallahydroquinone	C ₂₇ H ₄₀ O ₄	CDCl ₃	[123]
15-4-10	fallaquinone	C ₂₇ H ₃₈ O ₄	CDCl ₃	[123]
15-4-11	fallachromenoic acid	C ₂₇ H ₃₅ ClO ₄	CDCl ₃	[123]
15-4-12	2-[2'(<i>E</i>)-3',7',11',15'-tetramethylhexadec-en-1'-yl]-6-methyl-1,4-benzohydroquinone diacetate	C ₃₁ H ₅₀ O ₄	C ₆ D ₆	[124]
15-4-13	aplidic acid A	C ₂₃ H ₃₀ O ₄	CD ₃ OD	[125]
15-4-14	(3 <i>Z</i> ,11 <i>Z</i> ,7 <i>S</i> *,9 <i>S</i> *,10 <i>R</i> *)-9-acetoxy-6,13-dichloro-7,10-epoxypentadeca-3,11-dien-1-yne	C ₁₇ H ₂₂ Cl ₂ O ₃	CDCl ₃	[117]
15-4-15	(3 <i>Z</i> ,11 <i>Z</i> ,7 <i>S</i> *,9 <i>S</i> *,10 <i>R</i> *)-6-chloro-7,10-epoxypentadeca-3,11-dien-1-yne-9,13-diol	C ₁₅ H ₂₁ ClO ₃	CDCl ₃	[117]
15-4-16	(3 <i>Z</i> ,11 <i>Z</i> ,13 <i>E</i> ,7 <i>S</i> *,9 <i>S</i> *,10 <i>R</i> *)-9-acetoxy-6-chloro-7,10-epoxy-pentadeca-3,11,13-trien-1-yne	C ₁₇ H ₂₁ ClO ₃	CDCl ₃	[117]
15-4-17	(3 <i>Z</i> ,11 <i>Z</i> ,7 <i>S</i> *,9 <i>R</i> *,10 <i>R</i> *)-6-chloro-9,13-dibromo-7,10-epoxypentadeca-3,11-dien-1-yne	C ₁₅ H ₁₉ Br ₂ ClO	CDCl ₃	[117]
15-4-18	(3 <i>Z</i> ,11 <i>Z</i> ,7 <i>S</i> *,9 <i>R</i> *,10 <i>R</i> *)-13-methoxy-6-chloro-9-bromo-7,10-epoxypentadeca-3,11-dien-1-yne	C ₁₆ H ₂₂ BrClO ₂	CDCl ₃	[117]
15-4-19	(+)-muqubilone B	C ₂₄ H ₄₀ O ₆	CDCl ₃	[126]
15-4-20	—	C ₂₆ H ₄₀ O ₅	CDCl ₃	[121]
15-4-21	—	C ₂₆ H ₄₂ O ₅	CDCl ₃	[121]
15-4-22	—	C ₂₄ H ₃₈ O ₃	CDCl ₃	[121]
15-4-23	—	C ₂₄ H ₄₀ O ₃	CDCl ₃	[121]





**Table 15-4-2:** ^1H NMR spectroscopic data of marine natural products 15-4-1~15-4-3.

H	15-4-1	15-4-2	15-4-3
1	3.12 d(2.0)	3.13 d(2.0)	
2			2.47 m
3	5.59 dd(10.8, 2.0)	5.59 m	1.68 m, 1.39 m
4	6.06 dd(10.8, 7.3)	6.08 dt(10.7, 7.4)	1.37 m
5	2.70 m, 2.82 m	2.69 m, 2.60 m	1.44~1.52 m
6	4.04 ddd(10.3, 6.9, 2.9)	3.77 m	
7	5.01 ddd(10.3, 6.8, 3.4)	3.91 ddd(7.7, 6.8, 3.2)	1.44~1.52 m
8	2.49 ddd(14.7, 7.8, 6.8)	2.66 m, 2.52 m	2.52 m
9	5.32 m	5.61 m	1.16 d(6.9)
10	5.53 ddd(10.8, 7.3, 1.5)	5.73 m	1.22 s
11	2.77 m	3.21 d(7.1)	
12	5.37 m		
13	5.46 m	2.41 t(7.3)	
14	1.98 m	1.58 m	
15	0.94 t(7.3)	0.90 t(7.4)	
7'			2.03 s
8'			2.01 s
9'			2.00 s
OAc	2.08 s		
OMe			3.68 s

Table 15-4-3: ^1H NMR spectroscopic data of marine natural products 15-4-4~15-4-8.

H	15-4-4	15-4-5	15-4-6	15-4-7	15-4-8
1	3.29 d(7.1)	2.12 s	2.12 s	3.50 m	3.48 m
2	5.30 tq(5.1, 1.2)			1.76 quint(7.1)	1.59 quint(7.1)

Table 15-4-3 (continued)

H	15-4-4	15-4-5	15-4-6	15-4-7	15-4-8
3		2.40 dd(6.1~16.2) 2.21 m	2.39 dd(5.7~15.6) 2.21 dd(8.0~15.6)	2.34 q(7.1)	1.66 quint(7.1)
4	2.22 t(7.0)	1.97 m	1.98 m	6.95 dt(15.8, 7.1)	2.63 t(7.1)
5	2.41 m	1.28 m, 1.17 m	1.27 m, 1.15 m	6.14 d(15.8)	
6	6.60 m	1.29~1.30 m	1.29~1.31 m		6.12 d(15.8)
7		1.29~1.30 m	1.29~1.31 m	2.59 t(7.1)	6.93 dt(15.8, 7.1)
8		1.29~1.30 m	1.29~1.31 m	1.58 quint(7.1)	2.25 q(7.1)
9	5.32 d(9.8)	1.47 m	1.29~1.31 m	1.30 m	1.49 quint(7.1)
10	4.95 dm(9.8)	2.20 m	1.29~1.31 m	1.30 m	1.33 m
11		6.22 dt(15.3, 7.0)	1.63 m	1.30 m	1.30 m
12	1.71 (ov)	6.38 d(15.3)	2.60 t(7.4)	1.30 m	1.30 m
13	1.72 (ov)	0.89 d(6.5)	0.89 d(6.7)	1.30 m	1.30 m
14	1.82 d(0.8)			1.30 m	1.30 m
15	1.78 d(1.3)			1.30 m	1.30 m
16				0.90 t(6.9)	0.90 t(6.9)
1'				3.42 dd(10.0, 5.8)	3.42 dd(10.0, 5.8)
				3.48 m	3.47 m
2'		7.33 brd(7.0)	7.28 m	3.75 m	3.74 m
3'	6.53 d(2.8)	7.29 brt(7.3)	7.18 m	3.51 m	3.51 m
				3.57 dd(11.0, 4.8)	3.57 dd(11.3, 5.2)
4'		7.19 dt(7.4)	7.18 m		
5'	6.57 m	7.29 brt(7.3)	7.18 m		
6'	6.64 d(8.4)	7.33 brd(7.0)	7.28 m		

Table 15-4-4: ¹H NMR spectroscopic data of marine natural products 15-4-9~15-4-12.

H	15-4-9	15-4-10	15-4-11	15-4-12
1	3.28 d(7.0)	3.12 d(6.5)	1.67 s	3.23 d(7.1)
2	5.24 t(7.0)	5.14 m	2.12 m	5.3 t(7.1)
3			5.13 m	
4	2.09 m, 2.02 m	2.04 m		1.92
5	2.14 m	2.12 m	2.05 m	1.41 m, 1.26 m
6	5.07 m	5.10 m	2.56 q(7.5)	1.27 m, 1.22 m
7			5.89 t(7.6)	1.37 m
8	2.09 m, 2.02 m	2.05 m(ov)		1.28 m, 1.12 m
9	2.16 m	2.20 m	2.26 t(7.4)	1.22 m
10	5.50 t(7.5)	5.51 t(7.5)	2.00 t(7.4)	1.07 m
11			4.38 m	1.37 m
12	4.16 dd(5.5, 8.0)	4.16 dd(5.5)		1.33 m
13	2.44 m, 2.24 m	2.25 m, 2.43 m	4.89 m, 5.01 m	1.36 m, 1.21 m

Table 15-4-4 (continued)

H	15-4-9	15-4-10	15-4-11	15-4-12
14	5.09 m	5.09 m	1.81 s	1.13 m
15				1.48 dq(6.8)
16	1.65 s	1.64 s	1.57 s	0.86 d(6.8)
17	1.73 s	1.72 s	1.35 s	0.86 d(6.8)
18	1.75 s	1.62 s	2.13 s	0.86 d(6.8)
19	1.58 s	1.59 s		0.88 d(6.8)
20	4.27 d(1.5)	4.26 d(2.5)		1.51 s
3'	6.48 d(3.0)	6.45 br s	5.56 d(9.8)	6.98 d(2.4)
4'			6.24 d(9.8)	
5'	6.51 d(3.0)	6.54 br s	6.35 d(2.7)	6.77 d(2.4)
7'	2.18 s	2.05 s(ov)	6.50 d(2.7)	1.92 s
OAc				1.73 s, 1.79 s

Table 15-4-5: ¹H NMR spectroscopic data of marine natural products 15-4-13~15-4-15.

H	15-4-13	15-4-14	15-4-15
1		3.13 s(2.0)	3.13 d(1.8)
2	5.87 d(15.0)		
3	7.28 dd(15.0, 10.0)	5.61 dd(10.8, 2.0)	5.60 dd(10.8, 1.8)
4	6.30 dd(15.0, 10.0)	6.16 dt(10.8, 6.8)	6.14 dt(10.8, 7.0)
5	6.22 dd(15.0, 9.0)	2.81 m, 2.88 m	2.85 m
6	2.57 dd(9.0, 2.5)	4.01 ddd(9.3, 4.9, 4.4)	4.02 ddd(8.2, 5.0, 3.2)
7	1.61 m	4.32 ddd(7.3, 4.9, 2.0)	4.32 ddd(7.3, 5.0, 2.6)
8	1.23 td(11.5, 8.0)	α 2.53 ddd(14.2, 7.3, 6.8)	α 2.44 ddd(13.2, 7.3, 6.6)
	1.56 m	β 2.02 m	β 1.96 m
9	1.60~1.65 m, 1.70 m(ov)	4.91 ddd(6.8, 4.4, 2.4)	4.10 m
10	1.18 m, 1.86 m	4.82 m	4.69 dd(7.9, 6.4)
11	1.84 m	5.42 dd(10.8, 7.3)	5.46 dd(11.4, 7.9)
12	5.98 br d(10.0)	5.66 m	5.66 dd(11.4, 8.0)
13	5.52 dt(10.0, 3.5)	4.78 m	4.41 m
14	2.88 m	1.75 m, 1.82 m	1.52 m, 1.61 m
15	5.65 dd(15.0, 7.0)	0.99 t(7.3)	0.92 t(7.6)
16	6.01 dd(15.0, 10.0)		
17	6.07 dd(15.0, 10.0)		
18	5.60 dd(15.0, 7.5)		
19	2.13 dd(15.0, 7.5)		
20	1.70 m(ov)		
21	2.30 t(7.5)		
23	3.73 s		
OAc		2.06 s	

Table 15-4-6: ^1H NMR spectroscopic data of marine natural products **15-4-16~15-4-18**.

H	15-4-16	15-4-17	15-4-18
1	3.12 d(1.9)	3.14 d(2.0)	3.12 d(1.5)
3	5.60 dd(10.5, 1.9)	5.61 m	5.60 dd(10.2, 1.5)
4	6.16 dt(10.5, 6.7)	6.16 dt(10.5, 7.0)	6.15 dt(10.2, 7.0)
5	2.79 m, 2.89 m	2.91 m	2.90 m
6	4.01 ddd(9.3, 5.0, 4.4)	4.00 m	3.99 m
7	4.27 ddd(7.6, 5.0, 2.0)	4.62 m	4.60 m
8	α 2.52 ddd(14.0, 7.6, 6.7) β 2.05 m	α 2.51 dd(14.0, 6.4) β 2.72 m	α 2.53 dd(13.4, 6.1) β 2.73 m
9	4.98 m	4.47 m	4.46 m
10	4.94 m	4.82 dd(6.7, 3.5)	4.80 dd(7.9, 3.8)
11	5.19 dd(10.5, 8.2)	5.58 m	5.75 dd(10.5, 7.9)
12	6.10 m	5.70 dd(10.2, 10.2)	5.50 dd(10.5, 9.9)
13	6.40 m	4.57 m	3.80 m
14	5.76 ddd(13.7, 7.3, 7.0)	1.77 ddd(14.3, 7.3, 7.0) 1.86 ddd(14.3, 7.3, 6.7)	1.45 m 1.63 m
15	1.78 d(7.0)	0.98 t(7.3)	0.87 t(7.3)
OAc	2.06 s		
OMe			3.28 s

Table 15-4-7: ^1H NMR spectroscopic data of marine natural products **15-4-19~15-4-23**.

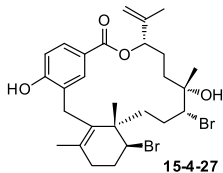
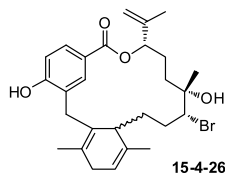
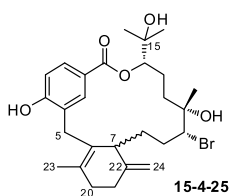
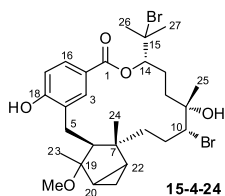
H	15-4-19	15-4-20	15-4-21	15-4-22	15-4-23
1	3.71 s(COOH)			1.34 s	1.35 s
2	2.55 quint(7.2)	2.89 d(15.3) 2.56 d(15.3)	2.90 d(15.4) 2.56 d(15.4)		
3	4.25 ddd(8.4, 7.2, 4.2)			2.02 m	2.00 m
4	ax 1.77 dtd (12.0, 8.4, 4.2) eq 1.70 dtd (12.0, 4.2, 3.0)	2.02 m	2.02 m	1.28 m, 1.46 m	1.28 m, 1.45 m
5	1.66 m	1.37 m, 1.67 m	1.35 m, 1.68 m		
6				1.43 m, 1.25 m	1.44 m, 1.24 m
7	1.49 m	1.44 m, 1.31 m	1.44 m, 1.27 m	1.63 m	1.64 m
8	2.04 m, 1.94 m	1.63 m	1.62 m	1.26 m, 1.15 m	1.27 m, 1.16 m
9	5.16 td(7.2, 1.2)	1.27 m, 1.14 m	1.27 m, 1.14 m	1.30 m	1.29~1.30 m
10		1.28 m	1.27 m	1.30 m	1.29~1.30 m
11	2.21 t(7.8)	1.30 m	1.29~1.30 m	1.30 m	1.29~1.30 m
12	2.56 t(7.8)	1.30 m	1.29~1.30 m	1.46 m	1.29~1.30 m
13		1.48 m	1.29~1.30 m	2.20 m	1.29~1.30 m
14		2.21 m	1.29~1.30 m	6.22 dt(15.7, 6.9)	1.26 m
15	1.51 m, 1.46 m	6.21 dt(15.9, 7.0)	1.62 m	6.38 d(15.7)	2.60 t(7.4)

Table 15-4-7 (continued)

H	15-4-19	15-4-20	15-4-21	15-4-22	15-4-23
16	1.42 m	6.39 d(15.9)	2.59 t(7.3)		
17	2.42 t(7.2)			7.33 br d(7.3)	7.26 m
18		7.33 br d(7.6)	7.26 m	7.28 brt(7.2)	7.17 m
19	2.14 s	7.28 brt(7.4)	7.17 m	7.18 brt(7.1)	7.17 m
20	1.20 d(7.2)	7.19 brt(7.2)	7.16 m	7.28 brt(7.2)	7.17 m
21	1.12 s	7.28 brt(7.4)	7.17 m	7.33 br d(7.3)	7.26 m
22	1.62 d(1.2)	7.33 br d(7.6)	7.26 m	0.97 d(6.5)	0.96 d(6.7)
23	1.13 s	0.99 d(6.6)	0.99 d(6.6)	1.39 s	1.39 s
24	1.13 s	1.39 s	1.38 s	0.93 d(6.7)	0.92 d(6.4)
25		0.92 d(6.5)	0.92 d(6.5)		
OMe		3.72 s	3.73 s		

Table 15-4-8: Compounds, MFs, and test solvents of marine natural products 15-4-24~15-4-34.

No.	Compounds	MFs	Test solvents	References
15-4-24	bromophycolide J	C ₂₈ H ₄₀ Br ₂ O ₅	CDCl ₃	[127]
15-4-25	bromophycolide K	C ₂₇ H ₃₇ BrO ₅	CDCl ₃	[127]
15-4-26	bromophycolide L	C ₂₇ H ₃₅ BrO ₄	CDCl ₃	[127]
15-4-27	bromophycolide M	C ₂₇ H ₃₆ Br ₂ O ₄	CDCl ₃	[127]
15-4-28	bromophycolide N	C ₂₇ H ₃₆ Br ₂ O ₄	CDCl ₃	[127]
15-4-29	bromophycolide O	C ₂₇ H ₃₇ Br ₃ O ₄	CDCl ₃	[127]
15-4-30	bromophycolide P	C ₂₇ H ₃₆ Br ₂ O ₄	CDCl ₃	[127]
15-4-31	bromophycolide Q	C ₂₇ H ₃₆ Br ₂ O ₄	CDCl ₃	[127]
15-4-32	neurymenolide A	C ₂₄ H ₃₂ O ₃	CDCl ₃	[128]
15-4-33	neurymenolide B	C ₂₆ H ₃₆ O ₃	CDCl ₃	[128]
15-4-34	hemi-phorboxazole A	C ₃₃ H ₄₂ N ₂ O ₇	CDCl ₃	[129]



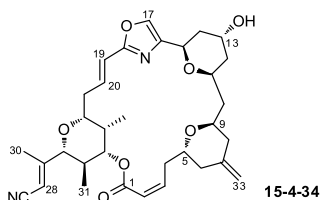
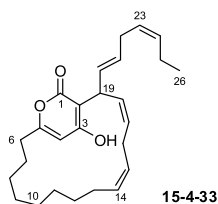
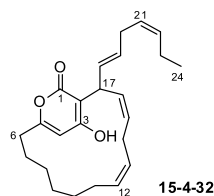
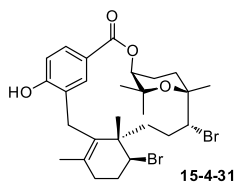
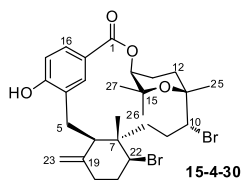
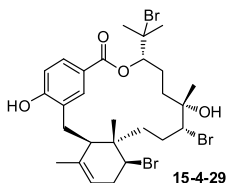
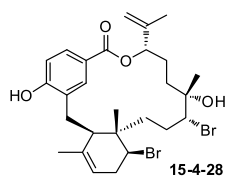


Table 15-4-9: ^1H NMR spectroscopic data of marine natural products 15-4-24~15-4-27.

H	15-4-24	15-4-25	15-4-26	15-4-27
3	8.11 br s	7.67 d(1.7)	7.67 d(1.9)	7.85 br s
5	2.37 d(15)	3.29 d(15)	3.22 d(15)	3.24 d(8.8)
	2.69 m	3.46 d(15)	3.46 d(15)	3.57 d(18)
6	2.59 m			
7		2.62 d(8.7)	3.41 m	
8	1.21 m, 2.62 m	1.45 m, 1.98 m	1.53 m, 1.97 m	1.58 m, 1.92 m
9	1.37 m, 1.75 m	1.44 m, 2.20 m	2.44 m, 2.50 m	1.93 m, 2.07 m
10	3.99 d(11)	3.96 m	3.97 dd(11, 3)	3.82 d(8.8)
12	1.50 m, 1.58 m	1.24 m, 1.46 m	1.67 m, 1.74 m	1.52 m, 1.77 m
13	2.11 m, 2.56 m	1.83 m, 1.94 m	1.73 m, 1.86 m	1.79 m, 1.83 m
14	4.88 d(11)	4.97 dd(2.7, 12)	5.52 br s	5.36 d(6.8)
16	7.81 d(8)	7.74 dd(1.8, 8.4)	7.73 dd(8.4, 1.9)	7.82 d(8.3)
17	6.78 d(8.2)	6.81 d(8.4)	6.78 d(8.3)	6.80 d(8.3)
20	1.55 m	2.24 m, 2.37 m	2.30 m, 2.42 m	2.13 m, 2.26 m
21	0.31 m	1.95 m	4.81 t(6.5)	2.25 m
	0.42 dd(5.8, 10)	2.17 m		2.36 m
22	1.14 m			4.49 dm(9.5)
23	1.38 s	1.91 s	1.89 s	1.41 s
24	0.55 s	4.46 s, 4.66 s	1.38 s	1.27 s
25	1.17 s	1.26 s	1.30 s	1.26 s
26	1.80 s	1.31 s	4.98 s, 5.07 s	4.92 s, 4.99 s

Table 15-4-9 (continued)

H	15-4-24	15-4-25	15-4-26	15-4-27
27	1.78 s	1.31 s	1.79 s	1.80 s
OMe	3.33 s			
OH	6.05 br s	5.48 br s	5.31 s	5.49 br s

Table 15-4-10: ¹H NMR spectroscopic data of marine natural products 15-4-28~15-4-31.

H	15-4-28	15-4-29	15-4-30	15-4-31
3	7.82 s	7.89 s	8.06 d(1.6)	7.64 s
5	2.60 d(15)	2.82 m	2.61 d(15)	3.15 d(17)
	2.83 dd(15, 9.0)	2.98 d(13)	3.17 dd(15, 8.0)	3.65 d(17)
6	2.67 m	2.84 m	2.37 m	
8	1.58 m, 2.18 m	1.23 m, 2.08 m	1.71 m, 2.13 m	1.58 m, 1.80 m
9	2.20 m, 2.39 m	1.98 m, 2.00 m	2.14 m, 2.58 m	2.05 m, 2.28 m
10	4.20 d(11)	3.79 d(11)	4.43 dd(11, 2.5)	4.46 dd(12, 2.5)
12	1.86 m, 2.02 m	1.53 m, 1.98 m	1.72 m, 2.35 m	1.69 m, 2.20 m
13	1.70 m, 2.05 m	2.13 m, 2.47 m	1.88 m, 2.30 m	1.80 m, 2.24 m
14	5.23 br s	5.00 dd(9.4, 3.4)	5.08 dd(6.6, 1.7)	4.94 d(7.0)
16	7.75 d(7.9)	7.74 d(8.3)	7.70 dd(8.3, 1.9)	7.77 d(8.5)
17	6.76 d(8.3)	6.81 d(8.3)	6.73 d(8.3)	6.80 d(8.5)
20	5.19 m	5.33 br s	1.97 m, 2.34 m	2.05 m, 2.20 m
21	2.53 m, 2.59 m	2.65 m	2.08 m, 2.22 m	2.20 m, 2.28 m
22	4.28 dd(11.0, 5.3)	4.24 dd(6.8, 6.2)	4.33 dd(12.0, 4.1)	4.57 d(10)
23	1.50 s	1.65 s	4.89 s, 5.30 s	1.34 s
24	1.06 s	0.89 s	0.99 s	1.34 s
25	1.40 s	1.32 s	1.47 s	1.42 s
26	4.84 s, 4.95 s	1.81 s	1.36 s	1.34 s
27	1.79 s	1.83 s	1.09 s	1.15 s
OH	5.33 br s	5.21 br s	5.68 br s	5.28 br s

Table 15-4-11: ¹H NMR spectroscopic data of marine natural products 15-4-32~15-4-34.

H	15-4-32	15-4-33	15-4-34
2			5.88 m
3			5.93 m
4	5.81 s	5.77 s	2.32 m, 3.46 m
5			4.17 m
6	2.30 ddd(14.0, 11.0, 3.3)	2.26 ddd(15.0, 11.0, 3.2)	2.05 br d(13.1)
	2.61 ddd(14.0, 10.0, 3.1)	2.63 ddd(15.0, 11.0, 3.4)	2.40 m
7	1.57 m, 1.86 m	1.49 m, 1.82 m	
8	1.24 m, 1.34 m	1.15 m, 1.22 m	1.86 m, 2.69 br d(12.3)
9	1.18 m, 1.35 m	1.08 m, 1.25 m	3.98 m

Table 15-4-11 (continued)

H	15-4-32	15-4-33	15-4-34
10	1.07 m, 1.15 m	1.24 m, 1.30 m	1.41, 1.86 m
11	1.76 m, 1.83 m	1.18 m, 1.23 m	4.06 m
12	5.23 m	1.20 m, 1.27 m	1.56 m, 1.68 br d(12.7)
13	5.21 m	1.88 m, 2.01 m	4.40 m
14	2.52 br d(17), 2.85 m	5.34 m	1.95 m
15	5.65 m	5.32 m	4.73 dd(11.0, 2.6)
16	5.64 m	2.63 m, 2.94 m	
17	4.55 br s	5.60 m	7.42 s
18	5.61 m	5.67 m	
19	5.61 m	4.61 br s	6.28 d(15.8)
20	2.77 m	5.62 m	6.64 ddd(16.0, 9.9, 6.4)
21	5.30 dt(11, 7.2)	5.62 m	2.40 m
			2.50 ddd(12.8, 10.2, 5.7)
22	5.42 dt(11, 7.2)	2.71 m	3.51 ddd(11.4, 5.1, 1.3)
23	2.00 p(15, 7.4)	5.33 m	2.30 m
24	0.93 t(7.4)	5.43 m	4.48 dd(11.2, 4.4)
25		1.99 m	1.95 m
26		0.94 t(7.5)	3.56 d(10.2)
28			5.37 s
30			2.09 s
31			0.75 d(6.4)
32			0.94 d(6.9)
33			4.60 br s, 4.97 br s
OH	6.48 br s	6.40 br s	

Table 15-4-12: Compounds, MFs, and test solvents of marine natural products 15-4-35~15-4-50.

No.	Compounds	MFs	Test solvents	References
15-4-35	bisglaucumlide A	C ₄₁ H ₆₀ O ₉	CDCl ₃	[130]
15-4-36	bisglaucumlide B	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[130]
15-4-37	bisglaucumlide C	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[130]
15-4-38	bisglaucumlide D	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[130]
15-4-39	bisglaucumlide E	C ₄₁ H ₆₀ O ₉	CDCl ₃	[131]
15-4-40	bisglaucumlide F	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[131]
15-4-41	bisglaucumlide G	C ₄₁ H ₆₀ O ₉	CDCl ₃	[131]
15-4-42	bisglaucumlide H	C ₄₁ H ₆₀ O ₉	CDCl ₃	[131]
15-4-43	bisglaucumlide I	C ₄₁ H ₆₀ O ₉	CDCl ₃	[131]
15-4-44	bisglaucumlide J	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[131]
15-4-45	bisglaucumlide K	C ₄₃ H ₆₂ O ₁₀	CDCl ₃	[131]
15-4-46	7 <i>E</i> -polymaxenolide	C ₃₈ H ₅₀ O ₈	C ₆ D ₆	[132]
15-4-47	7 <i>E</i> -5-epipolymaxenolide	C ₃₈ H ₅₀ O ₈	C ₆ D ₆	[132]
15-4-48	polymaxenolide A	C ₃₆ H ₄₈ O ₇	C ₆ D ₆	[132]
15-4-49	polymaxenolide B	C ₃₆ H ₄₈ O ₆	C ₆ D ₆	[132]
15-4-50	polymaxenolide C	C ₃₆ H ₄₈ O ₆	C ₆ D ₆	[132]

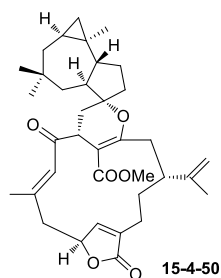
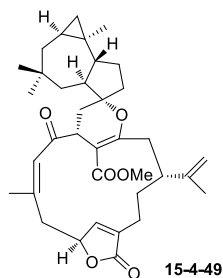
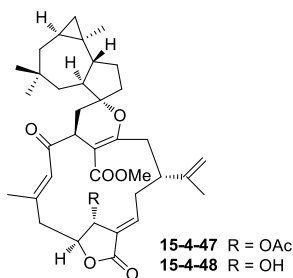
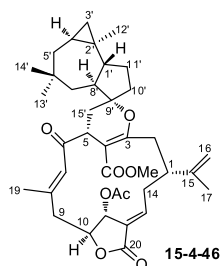
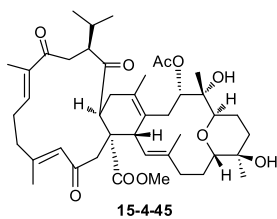
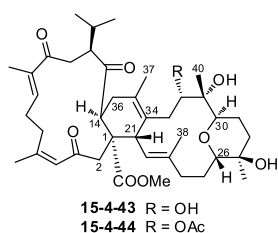
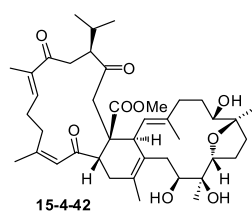
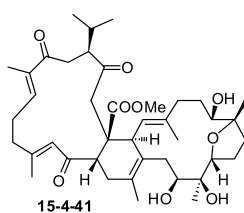
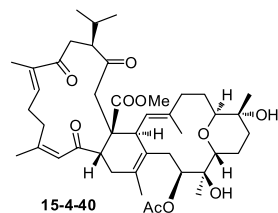
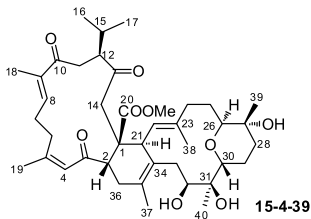
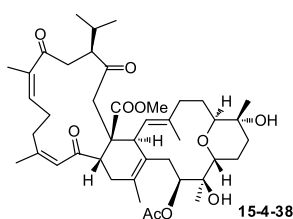
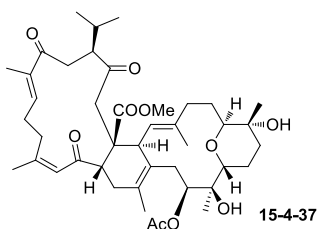
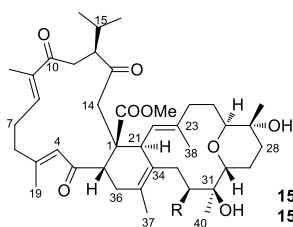


Table 15-4-13: ^1H NMR spectroscopic data of marine natural products **15-4-35**–**15-4-38**.

H	15-4-35	15-4-36	15-4-37	15-4-38
2	3.97 d(7.5)	3.78 d(7.8)	3.75 dd(3.6, 8.7)	3.42 (ov)
4	6.04 s	5.98 s	6.27 br s	6.20 s
6 α	2.42 (ov)	2.26 m	2.05 (ov)	2.72 (ov)
6 β	2.29 (ov)	2.37 (ov)	3.79 dt(2.9, 12.1)	2.08 (ov)
7	α 2.54(ov), β 2.42(ov)	α 2.51(ov), β 2.40(ov)	α 2.60 m, β 2.29 m	α 2.14(ov), β 2.38(ov)
8	6.24 m	6.22 dd(4.7, 6.8)	6.38 dd(5.7, 8.9)	5.50 t(7.7)
11 α	3.42 dd(5.3, 13.9)	3.44 dd(5.3, 13.9)	3.15 dd(7.6, 14.1)	3.13 dd(8.6, 18.6)
11 β	2.04 dd(4.9, 13.9)	2.02 dd(5.3, 13.9)	2.03 (ov)	2.24 dd(2.2, 18.6)
12	2.58 dd(5.3, 9.7)	2.51 m	2.70 (ov)	3.24 m
14 α	3.25 d(18.9)	3.23 d(19.0)	3.01 d(19.3)	2.88 d(18.4)
14 β	1.98 d(18.9)	2.12 d(19.0)	2.61 d(19.3)	2.95 d(18.4)
15	2.14 m	2.16 (ov)	2.03 (ov)	1.89
16	0.82 d(6.8)	0.80 d(6.9)	0.87 d(6.8)	0.83 d(6.8)
17	0.97 d(6.8)	0.95 d(6.8)	0.93 d(6.8)	0.93 d(6.8)
18	1.73 s	1.73 s	1.81 s	1.88 s
19	2.09 s	2.09 s	1.91 s	1.86 br s
21	3.29 d(11.6)	3.27 br d(11.7)	3.51 d(11.1)	3.41 (ov)
22	4.77 d(11.6)	4.78 d(11.7)	4.80 d(11.4)	4.85 d(11.1)
24 α	1.85 (ov)	1.85 t(3.6, 13.0)	1.86 m	1.86 (ov)
24 β	2.43 (ov)	2.44 m	2.43 dd(2.6, 10.2)	2.41 m
25	α 1.57 m, β 1.92 m	α 1.55(ov), β 1.95(ov)	α 1.57(ov), β 1.90(ov)	α 1.58(ov), β 1.85(ov)
26	3.64 br d(8.9)	3.68 d(10.2)	3.68 d(9.6)	3.69 m
28 α	1.69 (ov)	1.68 (ov)	1.68 m	1.70 (ov)
28 β	1.49 m	1.49 m	1.49 dt(5.1, 14.0)	1.48 dt(14.1, 4.9)
29	α 1.59 m, β 1.81 m	α 1.61(ov), β 1.70(ov)	α 1.59(ov), β 1.69(ov)	α 1.60(ov), β 1.70(ov)
30	3.68 br d(9.4)	3.65 (ov)	3.63 dd(2.1, 11.4)	3.63 br d(11.0)
32	3.56 (ov)	5.07 m	5.01 d(11.1)	5.04 d(10.9)
33 α	2.29 (ov)	2.15 (ov)	2.19 d(14.5)	2.24 (ov)
33 β	2.48 (ov)	2.74 m	2.70 (ov)	2.75 (ov)
36 α	1.87 d(18.1)	1.80 d(18.3)	2.02 (ov)	2.17 (ov)
36 β	2.93 br dd(7.2, 18.1)	2.83 br dd(7.7, 18.3)	2.70 (ov)	2.33 (ov)
37	1.71 s	1.67 br s	1.65 s	1.66 s
38	1.75 s	1.88 s	1.93 br s	1.95 br s
39	1.11 s	1.11 s	1.11 s	1.11 s
40	1.33 s	1.14 s	1.13 s	1.15 s
COOMe	3.57 s	3.55 s	3.54 s	3.54 s
OAc		2.09 s	1.95 s	2.04 s

Table 15-4-14: ^1H NMR spectroscopic data of marine natural products 15-4-39~15-4-42.

H	15-4-39	15-4-40	15-4-41	15-4-42
2	3.94 d(8.7)	3.69 d(8.5)	4.02 d(8.0)	3.63 brt(7.7)
4	6.22 s	5.66 s	6.05 br s	6.27 br s
6	3.86 m	2.15 m	2.41 (ov)	2.79 dt(10.2, 2.9)
	2.07 (ov)	2.08 (ov)	2.30 m	1.97 (ov)
7	2.57 dd(14.2, 8.7)	2.79 m	2.53 (ov)	2.37 m
	2.13 (ov)	2.40 m	2.41 (ov)	2.10 m
8	6.34 br t(7.3)	5.62 t(8.0)	6.24 m	5.51 t(8.2)
11	3.06 dd(14.2, 8.7)	3.05 dd(14.8, 10.9)	3.45 dd(13.7, 5.7)	3.12 dd(18.7, 9.2)
	2.09 dd(14.2, 3.1)	2.08 (ov)	2.01 dd(13.7, 5.1)	2.22 d(18.7)
12	2.76 m	2.80 dt(10.9, 3.3)	2.54 (ov)	3.25 m
14	2.38 d(19.3)	3.30 d(19.4)	3.30 d(19.1)	3.06 d(18.5)
	3.04 d(19.3)	2.36 d(19.4)	2.01 d(19.1)	2.86 d(18.5)
15	2.01 (ov)	2.07 (ov)	2.13 (ov)	1.91 m
16	0.89 d(6.7)	0.79 d(6.9)	0.80 d(6.8)	0.79 d(6.8)
17	0.92 d(6.7)	0.99 d(6.8)	0.96 d(6.8)	0.94 d(6.7)
18	1.80 br s	2.01 br s	1.73 br s	1.88 br s
19	1.92 br s	2.09 br s	2.09 br s	1.87 br s
21	3.64 (ov)	3.42 br d(11.6)	3.63 br d(11.0)	3.30 br d(10.6)
22	4.73 d(11.2)	4.74 d(11.6)	5.26 d(11.0)	5.16 d(10.6)
24	2.41 (ov)	2.44 m	2.46 (ov)	2.42 m
	1.86 (ov)	1.85 dt(3.4, 13.7)	2.13 (ov)	2.02 m
25	1.87 (ov)	1.95 (ov)	1.89 (ov)	1.98 (ov)
	1.56 (ov)	1.55 dt(3.4, 13.7)	1.65 (ov)	1.57 (ov)
26	3.64 (ov)	3.69 d(10.9)	3.38 br d(6.8)	3.39 br d(9.6)
28	1.69 (ov)	1.68 (ov)	2.09 (ov)	2.23 (ov)
	1.48 dt(4.3, 13.4)	1.49 dt(13.8, 4.5)	1.65 (ov)	1.62 m
29	1.72, 1.57	1.71, 1.59 m	1.89	2.23, 1.77
30	3.65 m	3.64 br d(9.5)	4.06 t(7.4)	3.99 dd(10.1, 5.7)
32	3.55 (ov)	5.07 br s	3.49 m	3.88 m
33	2.43 (ov), 2.29 (ov)	2.77 m, 2.14 m	2.48 (ov), 2.36 (ov)	3.03 m, 1.91 m
36	2.96 dd(18.4, 8.7)	2.86 br dd(18.3, 8.3)	2.79 dd(18.3, 8.0)	2.32 dd(18.7, 7.7)
	1.98 d(18.4)	1.74 br d(18.4)	1.93 br d(18.3)	2.18 m
37	1.69 br s	1.65 br s	1.68 br s	1.71 br s
38	1.81 br s	1.94 br s	1.74 br s	1.86 br s
39	1.12 s	1.11 s	1.15 s	1.15 br s
40	1.33 s	1.15 s	1.17 br s	1.11 br s
COOMe	3.54 s	3.52 s	3.57 s	3.56 s

Table 15-4-15: ^1H NMR spectroscopic data of marine natural products **15-4-43~15-4-45**.

H	15-4-43	15-4-44	15-4-45
2	3.15 d(19.0) 2.35 d(19.0)	3.10 d(19.2) 2.69 d(19.2)	2.92 br d(15.2) 2.56 br d(15.2)
3	6.07 s	6.07 s	5.55 br s
5	2.03 m	2.01 (ov)	2.38 m
6	3.79 dt(12.5, 2.6) 2.51 m	3.80 dt(2.5, 12.8) 2.55 m	2.48 m 2.09 m
7	2.25 m, 6.40 t(7.5)	2.21 m, 6.43 t(7.5)	6.61 br s
11	2.76 dd(15.7, 6.3) 2.61 dd(15.7, 5.7)	2.80 dd(16.2, 5.1) 2.38 dd(16.2, 5.7)	3.41 br s 1.86 (ov)
12	3.08 m	3.03 q(5.9)	
14	3.89 d(7.9)	3.62 (ov)	3.16 m
15	2.22 m	2.15 (ov)	2.16 m
16	0.87 d(6.8)	0.87 d(6.7)	0.69 d(6.5)
17	0.89 d(6.7)	0.87 d(6.7)	1.06 br s
18	1.69 br s	1.73 br s	1.75 br s
19	1.92 br s	1.90 br s	2.26 br s
21	3.58 br d(11.6)	3.63 (ov)	3.29 m
22	4.70 d(11.6)	4.75 d(11.5)	4.90 d(10.8)
24	2.43 (ov) 1.85 m	2.43 m 1.84 dt(3.0, 12.8)	2.42 (ov) 1.84 (ov)
25	1.87 (ov) 1.57 m	1.93 m 1.55 (ov)	1.81 (ov) 1.58 m
26	3.65 d(8.9)	3.68 d(9.9)	3.67 br d(8.3)
28	1.69 (ov) 1.48 dd(13.7, 4.5)	1.68 (ov) 1.49 dt(5.0, 13.9)	1.69 (ov) 1.47 dt(14.1, 4.7)
29	1.73 m, 1.57 (ov)	1.70 (ov), 1.57 (ov)	1.73 (ov), 1.61 m
30	3.64 d(12.7)	3.63	3.61 br d(10.1)
32	3.55 (ov)	5.06 d(11.4)	5.02 br d(10.8)
33	2.44 (ov) 2.27 br d(13.7)	2.71 (ov) 2.16 (ov)	2.76 br t(13.9) 2.20 br d(13.9)
36	3.12 m 1.93 (ov)	2.97 dd(18.7, 9.3) 1.94 (ov)	—
37	1.69 br s	1.64 br s	1.66 br s
38	1.82 br s	1.98 br s	1.91 br s
39	1.11 s	1.11 s	1.11 br s
40	1.31 s	1.13 s	1.14 s
COOMe	3.55 s	3.53 s	3.54 s
OAc		1.99 s	2.03 s

Table 15-4-16: ^1H NMR spectroscopic data of marine natural products 15-4-46~15-4-50.

H	15-4-46	15-4-47	15-4-48	15-4-49	15-4-50
1	2.91 brt(16)	2.95 brt(5.4)	2.92 brt(6)	2.72 brt(11.5)	2.54 brt(11.5)
2	2.10 brd(19)	2.22 br dd(14, 5.4)	2.22 br dd(14.5, 6)	1.95 t(12)	1.89 t(12)
	3.79 d(19)	4.48 brd(14)	4.47 d(14.5)	3.65 brd(12)	3.79 brd(12)
5	3.53 brd(8)	3.59 brd(3)	3.53 m	3.56 dd(12.2, 6.8)	3.59 dd(12, 7)
7	6.02 brs	6.05 brs	5.76 brs	5.77 brs	5.96 brs
9	1.80 m	2.36 dd(16, 6)	1.80 brd(16)	1.82 m	1.50 m
	2.28 m	2.60 d(16)	2.17 dd(16, 6)	2.04 m	2.29 m
10	4.22 t(12)	4.22 d(6)	4.19 d(6)	4.18 brd(7.5)	4.26 brt(5.4)
11	5.59 brs	5.71 brs	4.43 s	6.40 s	6.83 s
13	7.25 dd(17, 8.5)	7.56 d(8)	7.22 d(8.5)	2.04 m	2.06 m
				2.50 dd(16.0, 7.0)	2.60 dd(18, 12)
14	1.73 m	2.76 brd(20)	2.66 brd(18)	1.76 m	1.67 m
	4.05 t(18)	3.82 dt(20, 8)	3.76 dt(18, 6)	1.98 m	2.02 m
16	4.92 brs,	4.82 brs,	4.82 brs,	4.96 brs,	4.89 brs,
	4.94 brs	5.01 brs	4.99 brs	5.01 brs	4.95 brs
17	1.87 s	1.55 s	1.56 s	1.69 s	1.66 s
19	1.99 s	2.14 s	2.04 s	2.04 s	1.76 s
21	3.40 s	3.45 s	3.29 s	3.38 s	3.38 s
23	1.60 s	1.47 s			
1'	0.82 m	0.91 m	0.89 m	0.93 m	0.93 m
3'	0.15 t(6.6)	0.09 t(4.2)	0.03 t(4)	0.13 t(4.2)	0.14 t(4.2)
	0.55 dd(12.0, 6.6)	0.48 dd(8.6, 4.2)	0.43 dd(8.4, 4)	0.51 dd(7.5, 4.2)	0.53 dd(7.8, 4.2)
4'	0.44 m	0.36 m	0.30 m	0.39 m	0.38 m
5'	0.87 m	0.88 m	0.81 m	0.89 dd(14, 10.8)	0.85 dd(14.4, 10.8)
	1.76 m	1.68 brd(6)	1.62 dd(14, 6)	1.76 m	1.68 m
7'	0.74 t(18)	0.73 t(12.6)	0.66 t(12.5)	0.74 t(12.6)	0.74 t(12.6)
	1.51 m	1.44 m	1.40 brd(12.5)	1.76 m	1.40 m
8'	2.30 m	2.10 m	2.06 m	2.32 brt(10.0)	2.27 m
10'	1.22 m, 1.55 m	1.31 m, 1.76 m	1.26 m, 1.73 m	1.25 m, 1.54 m	1.26 m, 1.53 m
11'	1.13 m, 1.55 m	1.67 m, 1.76 m	1.54 m, 1.76 m	1.25 m, 1.58 m	1.55 m, 1.67 m
12'	0.98 s	0.96 s	0.90 s	0.93 s	0.93 s
13'	1.10 s	0.90 s	0.88 s	1.18 s	1.00 s
14'	0.94 s	0.84 s	0.78 s	1.10 s	0.82 s
15'	1.29 m	1.44 m	1.43 dd(13, 7.2)	1.53 m	1.39 m
	2.04 brd(8)	2.46 dd(13, 3)	2.38 d(13)	1.65 m	1.69 m

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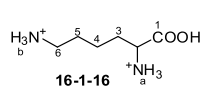
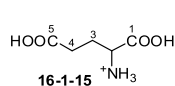
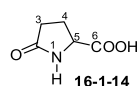
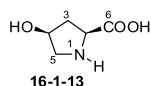
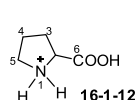
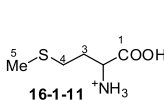
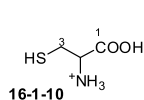
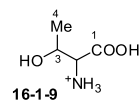
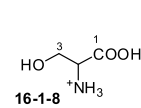
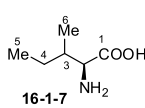
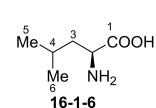
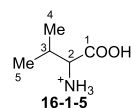
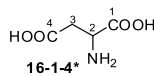
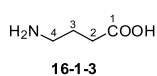
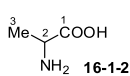
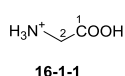
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16 Amino acids and sugars

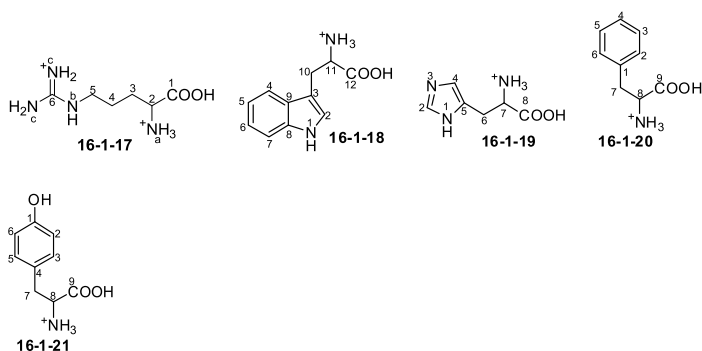
16.1 Amino acids

Table 16-1-1: Compounds, MFs, and test solvents of amino acids 16-1-1~16-1-21.

No.	Compounds	MFs	Test solvents	References
16-1-1	glycine	C ₂ H ₆ NO ₂	–	[1]
16-1-2	alanine	C ₃ H ₇ NO ₂	D ₂ O	[2]
16-1-3	4-aminobutyric acid	C ₄ H ₉ NO ₂	D ₂ O	[2]
16-1-4	asparaginic acid	C ₄ H ₇ NO ₄	D ₂ O	[3]
16-1-5	valine	C ₅ H ₁₂ NO ₂	–	[1]
16-1-6	leucine	C ₆ H ₁₃ NO ₂	CF ₃ COOD	[4]
16-1-7	isoleucine	C ₆ H ₁₃ NO ₂	CF ₃ COOD	[4]
16-1-8	serine	C ₃ H ₈ NO ₃	–	[1]
16-1-9	threonine	C ₄ H ₁₀ NO ₃	–	[1]
16-1-10	cysteine	C ₃ H ₈ NO ₂ S	–	[1]
16-1-11	methionine	C ₅ H ₁₂ NO ₂ S	–	[1]
16-1-12	proline	C ₅ H ₁₀ NO ₂	–	[1]
16-1-13	2S,4S-4-hydroxy proline	C ₅ H ₉ NO ₃	CD ₃ OD	[5]
16-1-14	pyroglutamic acid	C ₅ H ₇ NO ₃	CD ₃ OD	[6]
16-1-15	glutamic acid	C ₅ H ₁₀ NO ₄	–	[1]
16-1-16	lysine	C ₆ H ₁₆ N ₂ O ₂	–	[1]
16-1-17	arginine	C ₆ H ₁₆ N ₄ O ₂	–	[1]
16-1-18	tryptophan	C ₁₁ H ₁₃ N ₂ O	–	[1]
16-1-19	histidine	C ₆ H ₁₀ N ₃ O ₂	–	[1]
16-1-20	phenylalanine	C ₉ H ₁₂ NO ₂	–	[1]
16-1-21	tyrosine	C ₉ H ₁₂ NO ₃	–	[1]



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*Typographic error exists in the literature, drawing the substituent of COOH on C-1 as CHO.

Table 16-1-2: ^1H NMR spectroscopic data of amino acids 16-1-1~16-1-5.

H	16-1-1	16-1-2	16-1-3	16-1-4	16-1-5
2	4.28(5.7)	3.60 t(7.2)	2.84 t(7.6)(2H)	3.86 dd(4.2, 7.5)	4.32 (4.2, 5.7)
3		1.32 d(7.2)	1.73 m(2H)	2.8 dd(4.5, 16.8) 2.7 dd(7.8, 16.8)	2.60 (4.2, 6.9)
4			2.13 t(7.6)(2H)		1.25 (5.7, 6.9)
5					1.25 (5.7, 6.9)
NH ₂	7.47(5.7)				7.33

Table 16-1-3: ^1H NMR spectroscopic data of amino acids 16-1-6~16-1-10.

H	16-1-6	16-1-7	16-1-8	16-1-9	16-1-10
2	4.29 m	4.27 m	4.65 (4.0, 4.0, 6)	4.44 (4.5, 5.5)	4.68 (5.0, 5.0, 5.3)
3	1.84 m, 1.95 m	2.14 m	4.51 (4.0, 13.5) 4.56 (4.0, 13.5)	4.82 (4.5, 6.5)	3.36 (5.0, 15.5, 9.1) 3.41 (5.0, 15.5, 9.1)
4	1.79 m	1.41 m, 1.56 m		1.67 (6.5)	
5	0.96 m	0.96 m			
6	0.96 m	1.07 d(7)			
NH ₂			7.70 (6)	7.63	7.58(5.3)
SH					1.84 (9.1, 9.1)

Table 16-1-4: ^1H NMR spectroscopic data of amino acids 16-1-11~16-1-15.

H	16-1-11	16-1-12	16-1-13	16-1-14	16-1-15
2	4.67 (7.7, 4.4, 5.5)	4.78 (9.0, 7.0, 6.6, 4.4)	$\approx$ 5 (8.2, 10.4)		4.60 (5.6, 5.6, 5.5)
3	2.50 (7.7, 15.7, 6.5)	2.74 (9.0, 14.0, $\approx$ 6.8)	2.95 (8.2, 15.0)	2.33 m(2H)	2.63 (5.6, 15.5, 6.2)

Table 16-1-4 (continued)

H	16-1-11	16-1-12	16-1-13	16-1-14	16-1-15
	2.65 (4.4, 15.7, 6.5)	2.50 (7.0, 14.0, ≈6.8)	2.56 (10.4, 15.0)		2.55 (5.6, 15.5, 6.2)
4	2.96 (6.5, 6.5)	2.35 (≈6.8, ≈6.8) 2.29 (≈6.8, ≈6.8)	≈5 (<2, 4.2)	2.49 m, 2.16 m	3.01 (6.2, 6.2)
5	2.27	3.82, 3.76	3.9, 3.9	4.25 dd(4.0, 8.0)	
NH ₂	7.73 (5.5)	8.08 (6.6), 7.71 (4.4)			7.71 (5.5)

Table 16-1-5: ¹H NMR spectroscopic data of amino acids **16-1-16~16-1-18**.

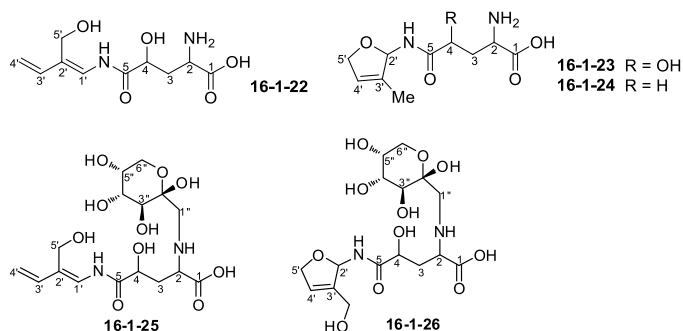
H	16-1-16	16-1-17	16-1-18
2	4.52 (5.6, 5.6, 5.8)	4.46 (5.3, 5.3, 5.5)	7.25
3	2.35 (5.6, ≈15, ≈6.0) 2.26 (5.6, ≈15, ≈6.0)	2.34 (5.3, ≈15, ≈6, ≈6) 2.25 (5.3, ≈15, ≈6, ≈6)	
4	≈1.8(≈6.0, ≈6.0)	≈2.08 (≈6, ≈6, 6.5) ≈2.00 (≈6, ≈6, 6.5)	
5	2.00(≈6.0)	3.43 (6.5, 6.5, 5.3)	
6	3.38(≈6.0, 6.0)		
10			3.79 (4.0, 15.5) 3.57 (8.0, 15.5)
11			4.71 (4.0, 8.0)
NH ₂	7.60 (NH ₂ -a) 6.97 (≈6.0)(NH ₂ -b)	7.60 (5.5)(NH ₂ -a) 6.50 (5.3)(NH ₂ -b) 6.19 (NH ₂ -c)	7.05

Table 16-1-6: ¹H NMR spectroscopic data of amino acids **16-1-19~16-1-21**.

H	16-1-19	16-1-20	16-1-21
2	8.73 (1.4)	7.3	7.03 (8.5)
3		7.4~7.5	7.27 (8.5)
4	7.66 (1.4)	7.4~7.5	
5		7.4~7.5	7.27 (8.5)
6	3.87 (6.4)	7.4~7.5	7.03 (8.5)
7	4.91 (6.4)	3.64 (4.5, 14.5) 3.37 (8.5, 14.5)	3.60 (4.5, 15.0) 3.34 (8.5, 15.0)
8		4.68 (4.5, 8.5)	4.64 (4.5, 8.5)
NH ₂	7.82	7.33	7.4

Table 16-1-7: Compounds, MFs, and test solvents of amino acids **16-1-22~16-1-26**.

No.	Compounds	MFs	Test solvents	References
16-1-22	pinnatanine	C ₁₀ H ₁₆ N ₂ O ₅	D ₂ O	[7]
16-1-23	longitubanine A	C ₁₀ H ₁₆ N ₂ O ₅	D ₂ O	[7]
16-1-24	longitubanine B	C ₁₀ H ₁₆ N ₂ O ₄	D ₂ O	[7]
16-1-25	kwansonine B	C ₁₆ H ₂₆ N ₂ O ₁₀	D ₂ O	[7]
16-1-26	kwansonine A	C ₁₆ H ₂₆ N ₂ O ₁₁	D ₂ O	[7]

**Table 16-1-8:** ¹H NMR spectroscopic data of amino acids **16-1-22~16-1-26**.

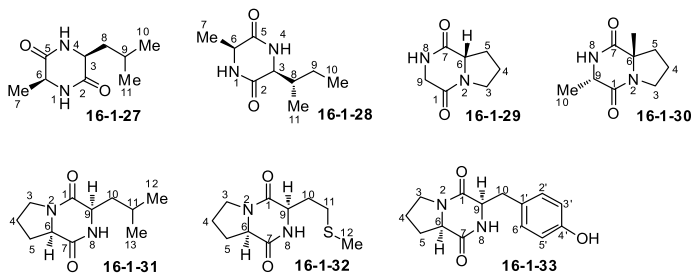
H	16-1-22	16-1-23	16-1-24	16-1-25	16-1-26
2	3.79 dd(3.7, 7.5)	3.95 dd(2.9, 6.3)	3.78 dd(6.2, 13.4)	3.80 dd(4.0, 6.0)	3.79 dd(3.8, 6.4)
3	2.09 m, 2.19 m	2.22 ddd(2.9, 8.1, 12.8) 2.33 ddd(2.9, 6.3, 12.8)	2.15 m(2H)	2.22 m	2.17 ddd(3.8, 6.4, 15.8) 2.24 ddd(3.8, 9.1, 15.8)
4	4.32 dd(3.0, 9.3)	4.33 dd(2.9, 8.1)	2.43 ddd(6.2, 6.5, 13.4) 2.49 ddd(6.2, 6.5, 13.4)	4.35 dd(4.3, 8.5)	4.22 dd(3.8, 9.1)
1'	6.61 brs			6.61 brs	
2'		6.21 m	6.18 brs		6.26 brs
3'	6.51 dd(11.3, 17.8)			6.51 dd(11.4, 17.7)	
4'	5.18 dd(1.1, 11.3)	5.89 ddd(2.0, 5.1, 6.5)	5.88 m	5.18 br d(11.4)	6.04 brs
	5.32 dd(1.1, 17.8)			5.32 br d(17.7)	
5'	4.18 s	4.55 ddd(2.0, 5.1, 10.6) 4.66 ddd(2.0, 5.1, 10.6)	4.51 ddd(2.0, 4.1, 12.7) 4.64 ddd(2.0, 4.1, 12.7)	4.17 s	4.49 d(13.0) 4.59 m

Table 16-1-8 (continued)

H	16-1-22	16-1-23	16-1-24	16-1-25	16-1-26
6'		1.73 brs	1.73 brs		4.04 br d(14.1) 4.10 br d(14.1)
1''				3.13 d(13.0) 3.27 d(13.0)	3.15 d(12.9) 3.26 d(12.9)
2''					
3''				3.65 d(10.0)	3.66 d(10.0)
4''				3.74 dd(2.8, 10.0)	3.75 dd(1.9, 3.4)
5''				3.86 m	3.87 dd(1.9, 3.4)
6''				3.61 br d(13.3) 3.88 d(13.3)	3.63 dd(1.9, 12.4) 3.90 d(12.4)

Table 16-1-9: Compounds, MFs, and test solvents of amino acids 16-1-27~16-1-33.

No.	Compounds	MFs	Test solvents	References
16-1-27	cyclo-(Ala-Leu)	C ₉ H ₁₆ N ₂ O ₂	C ₅ D ₅ N	[8]
16-1-28	cyclo-(Ala-Ile)	C ₉ H ₁₆ N ₂ O ₂	C ₅ D ₅ N	[8]
16-1-29	(cyclo-(Gly-Pro))dipeptide	C ₇ H ₁₀ N ₂ O ₂	DMSO- <i>d</i> ₆	[9]
16-1-30	(cyclo-(Ala-Pro))dipeptide	C ₈ H ₁₂ N ₂ O ₂	CDCl ₃	[10]
16-1-31	(cyclo-(Leu-Pro))dipeptide	C ₁₁ H ₁₈ N ₂ O ₂	CDCl ₃	[10]
16-1-32	cyclo-(L-Pro-L-Met)	C ₁₀ H ₁₆ N ₂ O ₂ S	CDCl ₃	[11]
16-1-33	cyclo-(L-Pro-L-Tyr)	C ₁₄ H ₁₆ N ₂ O ₃	CDCl ₃	[11]

**Table 16-1-10:** ¹H NMR spectroscopic data of amino acids 16-1-27~16-1-31.

H	16-1-27	16-1-28	16-1-29	16-1-30	16-1-31
1	9.30 s(NH)	9.28 s(NH)			
3	4.26 m	4.18 m	3.30 m(2H) ^①	3.60 m(2H)	3.6 m(2H)
4	9.25 s(NH)	9.02 s(NH)	1.79 m(2H) ^①	1.5~2.6 m	1.4~2.5 m

Table 16-1-10 (continued)

H	16-1-27	16-1-28	16-1-29	16-1-30	16-1-31
5			2.10 m, 1.79 m ^①	1.5~2.6 m	1.4~2.5 m
6	4.35 m	4.35 m	4.07 br d(7.9) ^①	4.0~4.3 m	3.90~4.24 m
7	1.69 d(6.9)	1.70 d(6.7)			
8	1.83 m, 2.06 m	2.28 m	8.05 (NH)		
9	2.07 m	2.06 m, 1.71 m	3.47 dd(16.4, 4.5) 3.95 d(16.4)	4.0~4.3 m	3.90~4.24 m
10	0.94 d(6.3)	0.86 t(7.4)		1.53 d	1.4~2.5 m
11	0.92 d(6.4)	1.13 d(7.1)			1.54 m(6.6) ^②
12					0.95 d(6.6) ^②
13					1.01(6.6) ^②

^① The data in the literature were not assigned, and here, the placements are only to display the data.

^② These data were from the same compound in [9].

Table 16-1-11: ¹H NMR spectroscopic data of amino acids 16-1-32 and 16-1-33.

H	16-1-32	16-1-33	H	16-1-32	16-1-33
3	3.57 m	3.57 m	10	2.38 m, 2.05 m	3.42 dd(11.0,14.0) 2.78 dd(9.6, 14.4)
4		1.85 m(2H)	11	2.69 t(7.0)	
5	2.43 m, 1.92 m	2.32 m, 1.96 m	12	2.14 s	
6	4.12 t(7.6)	4.07 t(7.6)	2', 6'		7.03 d(8.3)
8	6.55 br s(NH)	6.11 s(NH)	3', 5'		6.77 d(8.3)
9	4.24 t(5.2)	4.22 dd(2.9, 9.6)			

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16.2 Sugars

Table 16-2-1: Compounds, MFs, and test solvents of C₅-pyranoses 16-2-1~16-2-10.

No.	Compounds	MFs	Test solvents	References
16-2-1	α-xylose	C ₅ H ₁₀ O ₅	—	[12]
16-2-2	β-xylose	C ₅ H ₁₀ O ₅	—	[12]
16-2-3	α-lyxose	C ₅ H ₁₀ O ₅	—	[12]
16-2-4	β-lyxose	C ₅ H ₁₀ O ₅	—	[12]
16-2-5	α-arabinose	C ₅ H ₁₀ O ₅	—	[12]
16-2-6	β-arabinose	C ₅ H ₁₀ O ₅	—	[12]
16-2-7	α-ribose	C ₅ H ₁₀ O ₅	—	[12]
16-2-8	β-ribose	C ₅ H ₁₀ O ₅	—	[12]
16-2-9	methyl 2,3-anhydro-β-D-ribofuranoside	C ₆ H ₁₀ O ₄	—	[12]
16-2-10	methyl 2,3-anhydro-β-L-ribofuranoside	C ₆ H ₁₀ O ₄	—	[12]

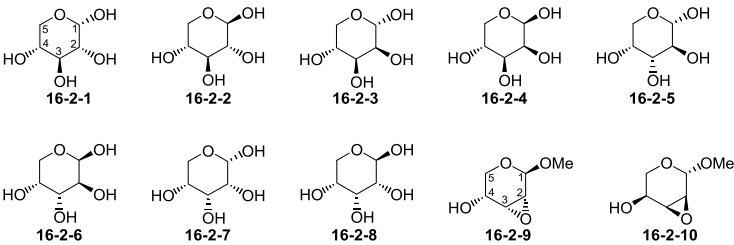


Table 16-2-2: ¹H NMR spectroscopic data of C₅-pyranoses 16-2-1~16-2-5.

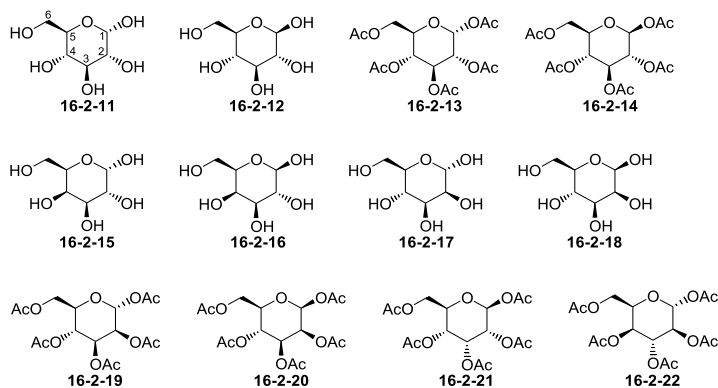
H	16-2-1	16-2-2	16-2-3	16-2-4	16-2-5
1	5.26 (3.1)	4.65 (7.4)	5.08 (4.2)	4.94 (1.5)	4.60 (7.2)
2	3.63 (3.1)	3.26 (7.4, 8.5)	3.90 (4.2)	4.04 (1.5, 3)	3.58 (9.9, 7.2)
3		3.49 (8.5, 8.5)			3.72 (9.9, 3.6)
4		3.58 (8.5, 10, 4.3)			4.04 (3.6)
5		3.36		3.32	
		4.00		4.09	

Table 16-2-3: ¹H NMR spectroscopic data of C₅-pyranoses 16-2-6~16-2-10.

H	16-2-6	16-2-7	16-2-8	16-2-9	16-2-10
1	5.34 (2.7)	4.91 (2.1)	4.99 (6.4)	4.84	4.85
2	3.93 (2.7)	3.85 (2.1)	3.60 (3.1, 6.4)	3.56 (4)	3.52 (3.8)
3	3.93 (3.4)		4.13 (3.1, 3.0)	3.20	3.17
4	4.07 (3.4)		3.79 (3.0, 10)	3.6~4.0	3.5~3.95
5				3.6~4.0 (2H)	3.5~3.95 (2H)

Table 16-2-4: Compounds, MFs, and test solvents of C₆-pyranoses **16-2-11~16-2-22**.

No.	Compounds	MFs	Test solvents	References
16-2-11	α -D-glucose	C ₆ H ₁₂ O ₆	—	[12]
16-2-12	β -D-glucose	C ₆ H ₁₂ O ₆	—	[12]
16-2-13	1,2,3,4,6-penta- <i>O</i> -acetyl- α -D-glucose	C ₁₆ H ₂₂ O ₁₁	—	[12]
16-2-14	1,2,3,4,6-penta- <i>O</i> -acetyl- β -D-glucose	C ₁₆ H ₂₂ O ₁₁	—	[12]
16-2-15	α -D-galactose	C ₆ H ₁₂ O ₆	—	[12]
16-2-16	β -D-galactose	C ₆ H ₁₂ O ₆	—	[12]
16-2-17	α -D-mannose	C ₆ H ₁₂ O ₆	—	[12]
16-2-18	β -D-mannose	C ₆ H ₁₂ O ₆	—	[12]
16-2-19	1,2,3,4,6-penta- <i>O</i> -acetyl- α -D-mannopyranose	C ₁₆ H ₂₂ O ₁₁	—	[12]
16-2-20	1,2,3,4,6-penta- <i>O</i> -acetyl- β -D-mannopyranose	C ₁₆ H ₂₂ O ₁₁	—	[12]
16-2-21	1,2,3,4,6-penta- <i>O</i> -acetyl- β -D-allopyranose	C ₁₆ H ₂₂ O ₁₁	—	[12]
16-2-22	1,2,3,4,6-penta- <i>O</i> -acetyl- α -D-idopyranose	C ₁₆ H ₂₂ O ₁₁	—	[12]

**Table 16-2-5:** ¹H NMR spectroscopic data of C₆-pyranoses **16-2-11~16-2-15**.

H	16-2-11	16-2-12	16-2-13	16-2-14	16-2-15
1	5.28	4.70	6.12 (3.6)	5.63 (8.0)	5.29
2			4.86 (3.6, 10.0)	4.9~5.2 (8.0, 10.2)	
3			5.17 (10.0, 9.3)	4.9~5.2 (10.2)	
4			4.89 (9.3, 9.3)	4.9~5.2 (9.5)	
5			3.92 (9.3, 4.7, 2.3)	3.94 (9.5, 4.6, 2.0)	
6			3.83, 4.15 ^①	4.09, 4.28 ^②	

^① $J_{5,6} = 4.7$, $J_{5,6'} = 2.3$, $J_{6,6'} = 12.2$.

^② $J_{5,6} = 4.6$, $J_{5,6'} = 2.0$, $J_{6,6'} = 12.5$.

Table 16-2-6: ^1H NMR spectroscopic data of C_6 -pyranoses 16-2-16~16-2-20.

H	16-2-16	16-2-17	16-2-18	16-2-19	16-2-20
1	4.63	5.20	4.92	5.81 (1.8)	5.75 (1.4)
2				5.0~5.23 (1.8)	5.33 (1.4, 3.2)
3				5.0~5.23	5.07 (3.2, 9.5)
4				5.0~5.23	5.12 (9.5, 9.3)
5				3.94 (4.8, 2.5)	3.70 (9.3, 5.0, 2.5)
6				3.97, 4.20 ^①	4.04, 4.22 ^②

^① $J_{5,6} = 4.8, J_{5,6'} = 2.5, J_{6,6'} = 12.5$.

^② $J_{5,6} = 5.0, J_{5,6'} = 2.5, J_{6,6'} = 12.2$.

Table 16-2-7: ^1H NMR spectroscopic data of C_6 -pyranoses 16-2-21~16-2-22.

H	16-2-21	16-2-22	H	16-2-21	16-2-22
1	6.05 (8.6)	5.98 (2.1)	4	5.05	4.91 (3.5, 2.1)
2	5.02 (8.6, 2.9)	4.81 (2.1, 3.6)	5	4.26	4.53 (2.1, 6.0)
3	5.75 (2.9, 2.9)	5.01 (3.6, 3.5)	6		4.15

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Index of compound names

- (+)-dihydrodiscorhabdin A 140
- (+)-discorhabdin X 140
- (+)-muquibilone B 153
- (-)-phorbasterone A 94
- 1,10-*seco*-withametelin B 16
- 1,2,3,4,6-penta-*O*-acetyl- α -D-glucose 176
- 1,2,3,4,6-penta-*O*-acetyl- α -D-idopyranose 176
- 1,2,3,4,6-penta-*O*-acetyl- α -D-mannopyranose 176
- 1,2,3,4,6-penta-*O*-acetyl- β -D-allopyranose 176
- 1,2,3,4,6-penta-*O*-acetyl- β -D-glucose 176
- 1,2,3,4,6-penta-*O*-acetyl- β -D-mannopyranose 176
- 1,2-didemethylcolchicine 33
- 1-carboxy-6-hydroxy-3,4-dihydro- β -carboline 140
- 1-carboxytryptargine 135
- 1-*epi*-10-hydroxydepressin 55
- 1-*epi*-10-oxo-11,12-dihydrodepressin 55
- 1-*epi*-10-oxodepressin 55
- 1-*epi*-depressin 55
- 1-*O*- β -D-glucopyranosyl-(2*S*,3*R*,4*E*,8*E*)-2-[(2-hydroxyicosanoyl)amido]-4,8-octadecadiene-1,3-diol 38
- 1-*O*- β -D-glucopyranosyl-(2*S*,3*R*,4*E*,8*Z*)-2-[(2*R*)-hydroxyicosanoyl]amido]-4,8-octadecadiene-1,3-diol 38
- 1-*O*- β -D-glucopyranosyl-(2*S*,3*R*,4*E*,8*Z*)-2-*N*-(2'-hydroxypalmitoyl)octadecaphinga-4,8-dienine 38
- 1-*O*- β -D-glucopyranosyl-(2*S*,3*R*,4*E*,8*Z*)-2-*N*-palmitoyl-octadecaphinga-4,8-dienine 38
- 10-hydroxydepressin 55
- 10-oxo-11,12-dihydrodepressin 55
- 11,12-epoxy-sarcophytoxide 59
- 11-acetoxy-3 β ,6 α -dihydroxy-24-methyl-9,11-*seco*-5 α -cholesta-7,24(28)-dien-9-one 95
- 11-acetoxy-3 β ,6 α -dihydroxy-9,11-*seco*-5 α -cholest-7-en-9-one 82
- 11 α ,19-dihydroxytelocinobufagin-3-(12-hydroxydodecanoic acid) ester 9
- 11 α ,19-dihydroxytelocinobufagin-3-(14-hydroxy-7-tetradecanoic acid) ester 9
- 11 α ,19-dihydroxytelocinobufagin-3-(14-hydroxytetradecanoic acid) ester 9
- 11 α -hydroxymexrenone 20
- 12-acetyl-13-*n*-butanoxyalcyopterosin D 49
- 12-acetyl-4-*n*-butanoxyalcyopterosin O 49
- 12-acetylalcyopterosin D 49
- 12-*n*-butanoxyalcyopterosin D 49
- 12 α -acetoxypetuniasterone M 20
- 12 β -hydroxy-1,10-*seco*-withametelin B 16
- 12 β -hydroxymexrenone 20
- 12 ξ -acetoxy-11 β -hydroxypetuniasterone D-7-acetate 20
- 12 ξ -acetoxy-11 β -hydroxypetuniasterone M-7-acetate 20
- 12*R*-hydroxycyclocitrinol 21
- 13-acetoxy-12-acetylalcyopterosin D 49
- 13-(*E*)-isogeoditin A 72
- 13-hydroxyalcyopterosin D 49
- 13-hydroxyisoprodysinoic acid 140
- 14-dehydroagrostistachin 55
- 14-methyleudistomidin C 138
- 14 β -hydroxypregn-5-enolone-3-*O*-sulfate 2
- 16- β -acetyloxy-pregn-4-17(20)-*trans*-dien-3-one 2
- 16-acetoxy-15-bromo-7-hydroxy-9(11)-parguerene 67
- 17-hydroxyagrostistachin 55
- 17 β -hydroxypetuniasterone A-7-acetate 20
- (17*R*,20*S*,24*R*)-17,25-dihydroxy-20,24-epoxy-14(18)-malabaricen-3-one 72
- (17*S*,20*R*,24*R*)-17,25-dihydroxy-20,24-epoxy-14(18)-malabaricen-3-one 72
- 19-hydroxyaraplysillin-I *N*²⁰-sulfanate sodium 140
- 19-oxobufalin 9
- 19-oxodesacetylcinobufagin 9
- 19-oxofasciospongine A 145
- 1 α ,3 β ,5 β ,11 α -tetrahydroxygorgostan-6-one 114
- 1 β -hydroxybufalin 9
- (1*R**,3*S**,4*R**,7*S**,8*S**,12*S**,13*S**)-7-formamidoamp-hilecta-11(20),14-diene 67
- (1*S*,3*S*,4*R*,7*S*,8*S*,11*S*,12*S*,13*S*,15*R*,20*R*)-7,20-diform-amidoisocycloamphilectane 67
- (1*S*,3*S*,4*R*,7*S*,8*S*,11*S*,12*S*,13*S*,15*R*,20*R*)-7-formamido-20-isocyanoisocycloamphilectane 67
- (1*S**,3*S**,4*R**,7*S**,8*S**,12*S**,13*S**)-7-formamidoamp-hilecta-11(20),15-diene 67
- (1*S**,3*S**,4*R**,7*S**,8*S**,12*S**,13*S**)-7-formamidocycloamphilect-11(20)-ene 67

- (15^{*},3Z,7Z,11S^{*},12R^{*})-18-hydroxydolabella-3,7-dien-2-one 59
- 2,3-didemethylcolchicine 33
- 2-[2'(E)-3',7',11',15'-tetramethylhexadec-2-en-1'-yl]-6-methyl-1,4-benzohydroquinone diacetate 153
- 2-*epi*-10-oxo-11,12-dihydrodepressin 55
- 2-methoxy-6-(methylethyl)cyclohepta-2,4,6-trien-1-one 33
- 2-methyl-9*H*-pyrido[3,4*b*]indole-3-carboxylic acid 134
- 2-methyleudistomin D 134
- 2-methyleudistomin J 134
- 20-*epi*-25 β -hydroxyverazine 20
- 20-*epi*-25 β -hydroxyverazine diacetate 20
- 20-*epi*-3-dehydroxy-3-oxo-5,6-dihydro-4,5-dehydroverazine 20
- 20-*epi*-4 β -hydroxyverazine 20
- 20-*epi*-4 β -hydroxyverazine triacetate 20
- 20-*epi*-verazine 20
- 20-*O*-methyl-24-*epi*-cyclocitrinol 20
- 20-*O*-methylcyclocitrinol 21
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